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Studies of gold-sulfur chemistry.

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STUDIES OF
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by

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ABSTRACT

The work in this thesis will be presented in two parts. The first part of this thesis deals with the interactions of gold(I) thiolates with serum albumin. The second portion of this thesis describes the synthesis and characterization of a gold-sulfur-molybdenum complex.

The first portion of this thesis examines the return of gold in biology and medicine for the treatment of rheumatoid arthritis (RA). Gold(I) thiolates have been used for chrysotherapy, but due to their adverse side effects, new gold(I) phosphine thiolates are being tested for use. However, the mechanisms of action of these drugs are as yet unknown.

The interaction of an analogue of Auranofin (triethylphosphinegold(I)chloride) with serum albumin were recorded by $^{31}P\{^{1}H\}$ nmr spectroscopy for a series of gold comlexes of PEt_3AuL and $[PEt_3AuL']^+ClO_4^-$ where L and L' are ligands containing biologically relevant donor atoms. This series of model compounds provides a ^{31}P nmr scale for the interaction of the 'PEt_3Au' moiety with proteins. The reactions of albumin and SH blocked albumin with PEt_3AuCl were monitored by ^{31}P nmr spectroscopy.

Comparison of the observed chemical shifts with those of the model compounds revealed preferential binding of gold to sulfur occurs. Fluorescence studies of the gold-protein interactions suggest that a protein conformational change may occur on binding of gold. The implications of these studies on the mechanism of action of anti-arthritic gold drugs is discussed.

The second portion of this thesis describes the synthesis, characterization and single crystal X-ray diffraction study of a gold-sulfur molybdenum complex for the purpose of studying a gold-sulfur bond.

The characterization of this complex, by $^{31}P\{^{1}H\}$ nmr and C, H analysis suggested the presence of a gold-sulfur-molybdenum bond. X-ray diffraction studies of this complex confirmed the proposed structure as being a trinuclear heterobimetallic complex. Although this complex is not directly related to the gold binding site observed in biological system, it does provide pertinent structural information about the nature of a gold-sulfur bond.

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To my father, and in loving memory of my mother.

For teaching me that

courage, patience and perseverence

makes anything possible.

This business is well ended.

Therefore, since brevity is the soul of wit...

...I will be brief.

Shakespeare,

(Polonius)

CHAPTER I

INTRODUCTION

Gold, is one of the most beautiful element, and has been known to man for thousands of years. In its elemental form, it is a soft ductile and malleable yellow metal.[1] It is normally found in alluvial deposits and seawater.[2] This metal, considered to be inert, can readily be dissolved by halide or sulfide ions in the presence of oxidizing agents to generate gold (III) or gold (I) complexes.[2] Its beauty and elusive nature render gold a highly prized metal for decorum (i.e.,jewellery) and for utilitarian value as legal monetary tender. [1] In ancient times gold was "considered by the wise to be able to remove the corruptions of the human body to such a degree that it could prolong life through the ages."[3]. The earliest recorded use of gold as a panacea dates back to the time of the ancient Chinese; 2500 BC.[3,4,10] It received much attention by well known physicians such as Marcus Varro and Pliney in the first century AD. Gold, physicians believed, assisted "wounded persons to ward off sorcerous curses, in ashen form to cure fistulas, discharges, putrid ulcers and sores, and boiled in honey as a liniment."[3] Perhaps the most famous cure-all

involving gold was available in the thirteenth century in the form of aurum potabile (gold dissolved in aqua regia and diluted with oil of rosemary or other essential oils) as a treatment for leprosy and other diseases.[3] However,it was not until 1890 that a complex of gold cyanide (AuCN) was discovered by Robert Koch to be an effective tuberculostat at concentrations of of 1:100,000.[4,4] Testing of this AuCN complex led him to the conclusion that it was much too toxic for human use. In order for these compounds to be of practical use, their toxicity had to be decreased.

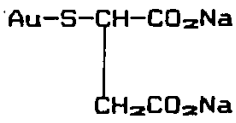
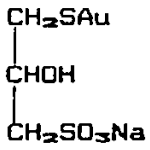
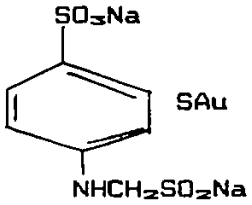
Intensive synthetic efforts by Lande in the early 1920's, accomplished this goal. Gold (I) thiolates were introduced for the treatment of tuberculosis.[7] (See Table 1.1) Patients treated with these gold (I) thiolates demonstrated good recovery rates from tuberculosis. He also noted that patients afflicted with rheumatoid arthritis as well as tuberculosis exhibited a remission of both diseases. It was on this basis that he incorrectly assumed a direct relationship between the tuberculosis bacilli and the cause for rheumatoid arthritis.[3]

Jacques Forestier and other physicians realized the potential for the use of gold drugs in the treatment of

TABLE 1.1

Gold (I) Thiolates

used in the treatment of Rheumatoid Arthritis

COMPOUND	TRADE NAME	STRUCTURE
Sodium Aurothiosulfate	Sanochrysin	$Na_3Au(S_2O_3)_2$
Sodium Aurothiomalate	Myochrysin	
Sodium Aurothiopropanol	Allochrysin	
Aurothioglucose	Solganol-B Oleosum	

rheumatoid arthritis, and assisted in popularizing these treatments in the Gold Decade (1925-1935).[4,10] During the late 1930's, interest in gold treatments waned with the increasing reports of the toxic side effects. These side effects such as nausea, vomitting, diarrhea, agranulocytosis, bone marrow depression and tissue necrosis in the kidney (due to retention of gold)[8] led the Empire Rheumatism Council in 1960 to perform controlled trials to determine to what extent the gold(I) thiolates could function effectively.[4,10] These studies concluded that 65% of the patients treated with these gold(I) thiolates, experienced complete remission of rheumatoid arthritis and only 35% of the patients treated experienced these harmful side effects.[4,8,11] Administration of these drugs is in the form of intramuscular (IM) injection. The dosage of 50 mg per week was stopped after 20 weeks to eliminate these side effects.[4] Ocassionally cessation of treatments caused a recurrence of the disease. As a result, a search was conducted for less toxic compounds which could be more efficiently controlled. This search led workers at Smith, Klein , French laboratories in Philadelphia in 1972 to test a variety of gold(I)phosphinethiolates for anti-inflammatory activity.[8] (See Table 1.2) Comparative studies of gold(I) thiolates

TABLE 1.2

GOLD (I) PHOSPHINE THIOLATES

1) $R_3P-AuCl$

R = Me, Et, i-Pr, n-Bu
Best results with R = Et

2) $R_3-P-Au-S-CH(CO_2H)-CH_2CO_2H$

R = alkyl, alkoxyl, phenyl

3) $R_1R_2P-(CH_2)_n-S$ bridging Au, Au, $S-(CH_2)_n-PR_1R_2$

(best results with n=2, R_1, R_2 = C_1 to C_2 alkyl)

4) (2,3,4,6-Tetra-O-Acetyl-1Thio-B-D-Glucopyranosato-S) (Triethyl phosphine)Gold. $C_{20}H_{34}AuO_9PS$*

See Figure 1.2

* This compound now undergoing clinical testing in the United Kingdom.

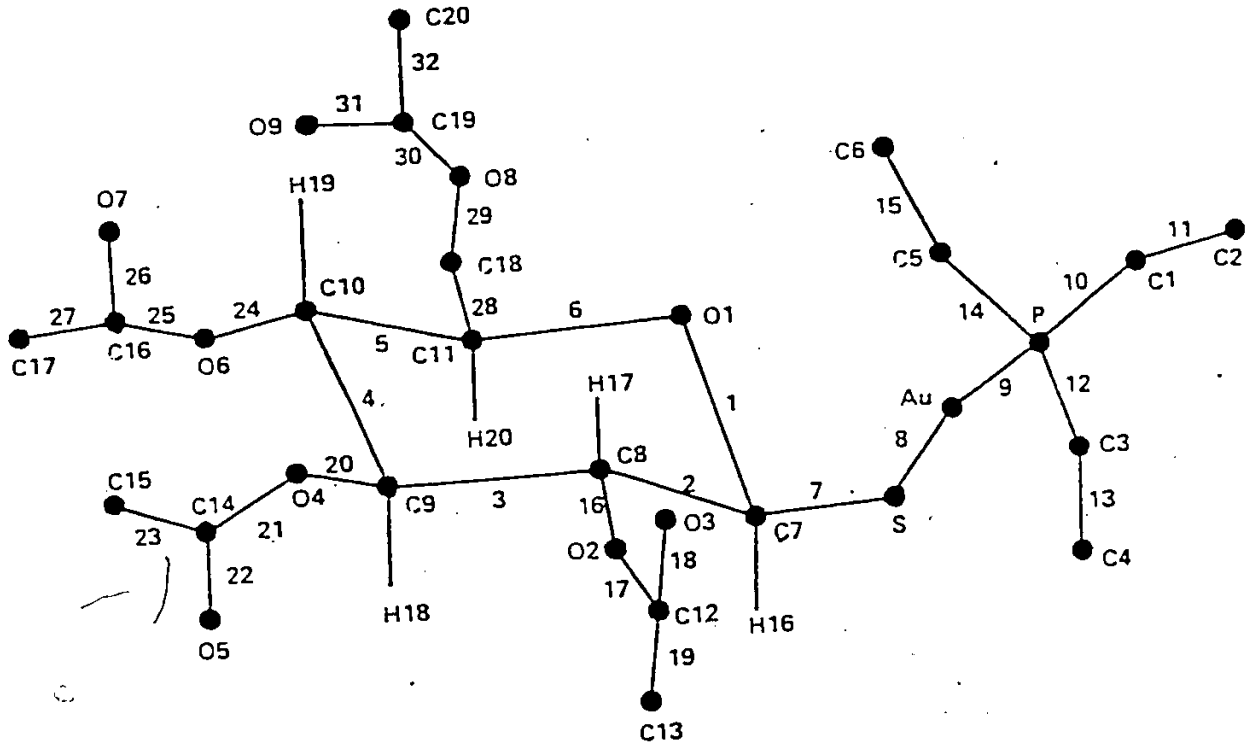


Fig. 1.2

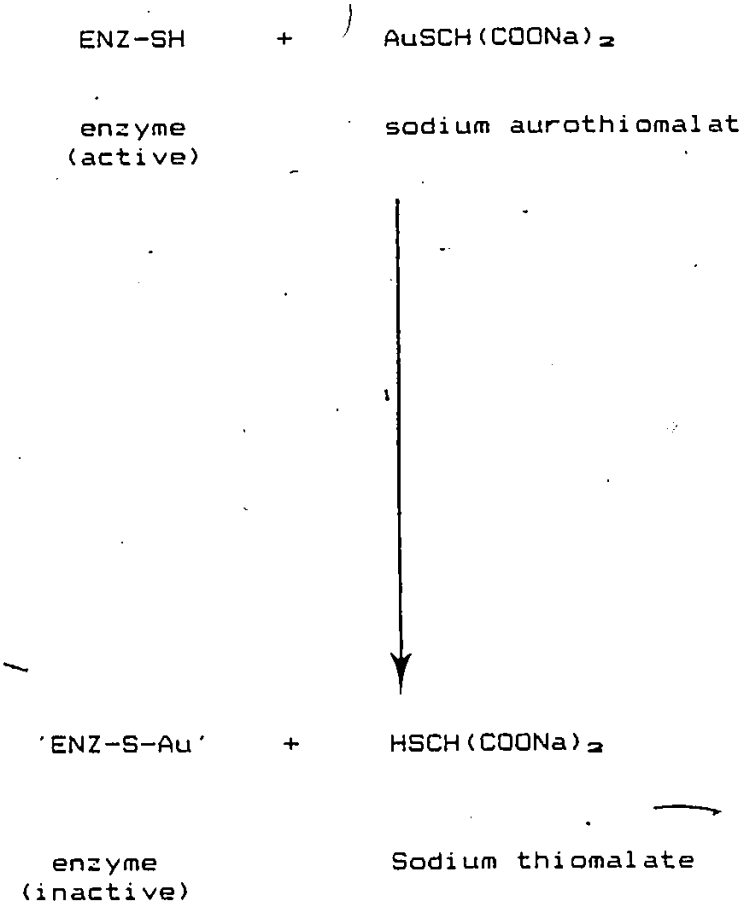
Crystal Structure of (2,3,4,6-Tetra-O-acetyl-1-Thio-B-D glucopyranosato-S-(triethyl phosphine) gold.[13]

$C_{20}H_{34}AuO_9PS$

(i.e., Myochrysin) and gold(I) phosphine thiolates (i.e, Auranofin) for use as anti-inflammatory agents were conducted. These studies concluded that the effects on inflammatory rheumatoid arthritis were not significantly different for the two drugs.[11] The gold(I) thiolates were thought to interfere with the inflammatory process by blocking or inhibiting some enzymatic ativity. Binding to the cysteine (SH) residue of the protein could occur by the exchange reaction in figure 1.1 [11] The exact enzyme system affected is unknown. A result of the exchange process occurs when the enzyme or protein is converted to the gold mercaptide, which presumably either blocks or inhibits its activity. In the case of gold(I)phosphinethiolates, it is suspected that the alkyl phosphine substituent increases the lipid solubility of the drug which allows it to cross the membrane and increase the serum gold level.[12] It has been suggested that up to 90% of the Auranofin is bound to the albumin.[4,11,12] One might speculate that the albumin acts as a transport agent, carrying the gold to the site of action. However, the nature of the gold-protein interaction is unknown. In this work I have attempted to identify a possible mechanism of action of this drug with albumin. This was accomplished by, using a compound

Figure 1.1

Proposed Scheme for gold (I) thiolate-protein binding



related to Auranofin.

Triethylphoshinegold(I)chloride was reacted with a variety of biologically relevant donor ligands and their ^{31}P nmr spectra recorded. By comparision of these spectra with the spectra of the triethylphosphinegold(I) moiety bound to albumin, a definite binding site of gold in albumin was determined. Fluorescence studies of the gold-protein complexes were used to examine the effect of gold binding to the SH residues.

The second part of this thesis shows the uses of tetrathiometalates as ligands in complex formation. The applications of thiometalates as ligands are well reviewed by A. Muller[13,14]. These tetrathiometalate ligands play a dominant role in bioinorganic systems due to their relevance in the electron transfer process.[14] MS_4^{2-} ligands are precursors for the synthesis of model compounds for the active site of a number of enzymes including Nitrogenase. The chalcogenmetalates can be prepared quite easily by the reaction of H_2S with an aqueous solution of the corresponding oxometalates.. These thiometalates are not very stable in solution and are distinguished by their high intensity absorption bands in the UV-vis region.

An examples of this type of bonding is given below.

$$[Cl_2Fe(MoS_4)]^{2-} + [FeCl_2] \longrightarrow [Cl_2Fe(MoS_4FeCl_2]^{2-}$$

MS_4^{2-} ligands are usually coordinated via a S to soft cations such as Cu^+, Ag^+, Au^+ and few are known due to their tendency to polymerize.[18].

In the second portion of this thesis, a gold-sulfur molybdenum complex was prepared and characterized employing MoS_4^{2-}. The reaction of tetrathiomolybdates with triethylphosphinegold(I)chloride was performed anaerobically. Single crystal X-ray diffraction studies confirmed the presence of a gold-sulfur-molybdenum bridge. This bridged complex demonstrates the possibility of metal-metal interactions which imply the possibility of unique reactivity patterns.

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CHAPTER II

A ^{31}P Nuclear Magnetic Resonance and Fluorescence Study of an Anti-Arthritic Gold Phosphine Drug with Albumin. A Bioinorganic Approach

2.2 INTRODUCTION

Recent reviews of gold chemistry have focused on applications in biology and medicine.[19,25] Of particular interest has been the use of gold thiolates in the treatment of rheumatoid arthritis (RA).[19,22,25] Side effects with these drugs can be severe, nonetheless, the ability of chrysotherapy (gold treatments) to cause remission of the disease results in these drugs being one of the standard prescriptions of modern medicine for severe cases of RA. The past decade has seen significant progress in reducing adverse side effects by the introduction of new gold-phosphine drugs.[22,24,27] These drugs are readily absorbed through the gut; thus they have the additional benefit of oral administration, in lieu of the painful intramuscular injections that are required with the gold-thiolate drugs.

The mechanism of action has been the subject of much speculation. Generally, many of the proposals deal with the alteration of enzyme function by gold complexation; however, the exact enzyme system or systems involved are unknown.[22] Initial in vivo studies investigating gold distributions throughout the body following drug administration revealed that between 50-90% of the gold present is bound to the serum protein, albumin.

The interactions of gold-phosphine drugs and albumin were investigated employing a bioinorganic approach.[20] Firstly, $^{31}P\{^{1}H\}$ nmr spectroscopy was used to examine an extensive series of model compounds designed to mimic the interactions of the 'PEt_3Au' moiety with biologically relevant donor ligands. Secondly, $^{31}P\{^{1}H\}$ nmr studies of the reactions of PEt_3AuCl with albumin were undertaken. This two phase approach provided a basis for conclusions regarding the nature of the gold binding to the protein. Effects of gold binding on protein conformation were investigated by fluorescence spectroscopy. The results of these studies are the subject of this thesis.

2.2 EXPERIMENTAL

Preparations of all gold compounds were performed under an atmosphere of dry, O_2 free N_2. $^{31}P\{^1H\}$ nmr spectra were recorded at 25°C on a Bruker CXP-100 pulse nmr spectrometer operating at 36.4 MHz with broad band proton decoupling. The $^{31}P\{^1H\}$ nmr chemical shifts are reported relative to 85% H_3PO_4. All fluorescence data were recorded on a locally assembled fluorometer consisting of an Oriele Xenon lamp, McPherson emmision monochromator and a Heath exciting monochromator. All ligands were purchased from the Aldrich Chemical Co. or Sigma Chemicals. The compound, $HAuCl_4$ was obtained on loan from Johnson-Matthey Ltd. Human Serum Albumin (HSA) and Bovine Serum Albumin (BSA) were purchased from Sigma Chemicals.

2.2.1 Preparation of Gold Compounds

i) Triethylphosphinegold(I)chloride was prepared from $HAuCl_4$ according to the literature methods of Mann, Wells and Purdie.[29] Et_3PAuCl was prepared by the addition of freshly distilled triethylphosphine (5g, 2 mol) in alcohol (20mL) to a well cooled, agitated mixture of chloroauric acid (20mL:, 0.95 mol) and alcohol (20mL). The mixture was stirred for one hour and then it was diluted

with 50mL water. The white product was collected, washed with water and dried. The melting point was determined to be 78°C which corresponded exactly to the literature value.[29] $^{31}P\{^1H\}$ nmr spectroscopy showed the compound to give a clean singlet at 31.7 ppm in $CDCl_3$.

ii) Et_3PAuCl_3 can be isolated as an intermediate product in the reduction of tetrachloroauric acid by triethylphosphine. It can also be prepared by the method of F. G. Mann and D. Purdie as follows.[30]. PEt_3AuCl is oxidized with Cl_2 in a solution of carbon tetrachloride as follows.

$$[AuCl(PEt_3)] + Cl_2 \longrightarrow [AuCl_3(PEt_3]$$

In this work, Et_3PAuCl_3 was isolated as an intermediate. C, H analysis,and melting point confirmed the compound to be of the same composition as the compound which was prepared by the aforementioned literature procedure.

iii) $(Et_3PAu)_2S$ was prepared according to the literature methods of Kowala and Swan.[31] A chloroform solution of triethylphosphinegold(I)chloride was shaken with aqueous sodium sulfide, thereby producing the yellow crystalline product.

iv) Preparation of Et_3PAuL

All compounds 2-18 were prepared using methods analogous to those published in the literature.[32,34] In some cases NBu_4OH (1M in methanol) was used in lieu of $NaOCH_3$ as the base. A sample preparation is given below.

Et_3PAuCl (50mg., 0.143mmol.) was dissolved in methanol (10 mL). Separately C_6H_5SH (15.7mg) was added to one equivalent of $NaOCH_3$ solution. The two solutions were combined and stirred for one hour. The solvent was removed, the residue was dissolved in $CDCl_3$ and the NaCl was removed by filtration through celite. The $CDCl_3$ contained pure $Et_3PAuSC_6H_5$ as shown by the clean sharp singlet in the $^{31}P\{^1H\}$ nmr spectrum.

v) Preparation of $Et_3PAuL^+ClO^{4-}$

All compounds 19-28 were prepared using routes analogous to literature methods.[35] A sample preparation is given. Et_3PAuCl (50mg, 0.143mmol) was dissolved in methanol (10mL). To this solution $AgClO_4$ (29.4mg) was added. After stirring for 5 minutes, the flocculent precipitate of AgCl was removed by filtration through celite. $C_6H_5NH_2$ (13.3 mg, 0.143 mmol) was added to the filtrate. The solvent was removed and replaced with

$CDCl_3$. $^{31}P\{^1H\}$ nmr spectral data showed a clean singlet indicative of pure $Et_3PAu(H_2NC_4H_8)^+ClO_4^-$.

2.2.2 i) Preparation of Albumin, HSA and BSA

0.100 g of the protein was dissolved in deionized, distilled water. Mercaptoethanol ($HSCH_2CH_2OH$, (14.25M, 70 μL) was added and stirred for 1/2 hour. The solution ws dialyzed against 4L of deoxygenated 50mM Tris(hydroxymethyl)aminomethane (TRIS) buffer (pH = 8.5). the protein was lyophilized and stored under N_2 at 0°C.

ii) Preparation of blocked albumin, HSA and BSA.

The protein was reduced as above. Iodoacetamide (100 mol excess) was added. The pH was raised to 8.5 and the solution was stirred for 1 hour. The solution was dialyzed against 8L of cold water, lyophilized and stored under N_2 at 0°C. See Figure 2.1.

2.2.3 Reactions of HSA, BSA, with Et_3PAuCl

The protein, (67 mg/mL solution, 50 μL was diluted with of D_2O (1.0 mL). To this solution, Et_3PAuCl (50 μL of 0.48M) in methanol was added. These solutions, were observed to be cloudy; and were centrifuged. $^{31}P\{^1H\}$ nmr data and fluorescence data were recorded for the

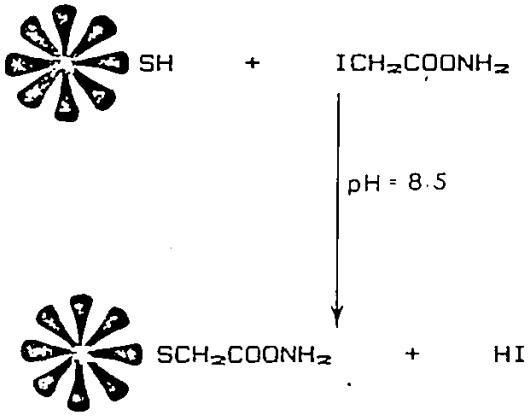


Figure 2.1

Preparation of blocked Serum Albumin

resulting solutions. Precise protein concentrations in the supernatants were determined to be 2.92 mg/mL, using the Bio-Rad Coomassie Blue Microprotein assay as developed by M. Bradford.[34]

2.3 RESULTS AND DISCUSSION

2.3.1 Model Compounds:

These linear gold (I) complexes of formulae PEt_3AuL and $[PEt_3AuL']^+ClO_4^-$, were prepared according to the following equations. The former neutral complexes where L is either thiolate, phenolate and carboxylate were prepared by the simple nucleophilic displacement of chloride from PEt_3AuCl [32,33] to generate a neutral complex. (See Eqn. 1, Fig 2.2a)

Both phenolate and carboxylate were used to mimic two different types of oxygen binding sites in biological systems. The latter linear gold (I) complexes were prepared by removal of the chloride by $AgClO_4$ followed by the addition of a neutral group V or VI donor molecule to generate the perchlorate salt. (See Eqn. 2, Fig 2.2b)

All of the gold compounds were characterized by $^{31}P\{^1H\}$ nmr spectra in chloroform. The purity of the complexes was indicated by the presence of only clean, sharp singlets in the $^{31}P\{^1H\}$ nmr spectra. Observed chemical shifts relative to 85% H_3PO_4 are summarized in Table 2.1.

Table 2.1 ^{31}P nmr data for gold compounds

(i) Complexes of the form PEt_3AuL

L	δ(ppm)[a]	L	δ(ppm)[a]
1. Cl^-	31.7	10. $EtO_2CCH(NH_2)CH_2S^-$	35.4[b]
2. PhO^-	27.2	11. $HO(CH_2)_2S^-$	37.9
3. $CH_3CO_2^-$	26.9	12. $Na^+O_2CCH_2S^-$	38.2
4. $CH_3O_2C(NHCOCH_3)CHCH_2C_6H_4O^-$	27.4[b]	13. $EtO_2CCH_2S^-$	37.8
5. $H_2NCH_2CO_2^-$	27.6	14. $NBu_4^{+-}O_2CCH(NH_2)C(CH_3)_2S^-$	37.8[b]
6. PhS^-	37.1	15. $C_{14}H_{19}OS^-$	36.0[b]
7. BuS^-	37.6	16. $C_6H_{11}O_5S^-$	37.5[b]
8. $^-S(CH_2)_2S^-$	36.1	17. $NBu_4^{+-}O_2CCH(CH_2CO_2^-NBu_4^+)S^-$	36.2[b]
9. o-$H_2NC_6H_4S^-$	37.3	18. $(CH_3)_2NCS_2^-$	34.1

(ii) Complexes of the form $PEt_3AuL^+ClO_4^-$

19. $EtNH_2$	30.6	22. $CH_3O_2CCH(NH_2)CH_2C_3H_3N_2$	29.9[b]
20. $PhNH_2$	31.2	23. $CH_3O_2CCH(NH_2)CH_2CH_2SCH_3$	24.3[b]
21. $C_3H_4N_2$	30.0[b]	24. Bu_2S	24.7
		25. none	27.7[b]

(iii) Others

26. PEt_3AuCl_3	54.5	27. $(PEt_3Au)_2S$	33.0

[a]Spectra were recorded in $CDCl_3$ at 25°C

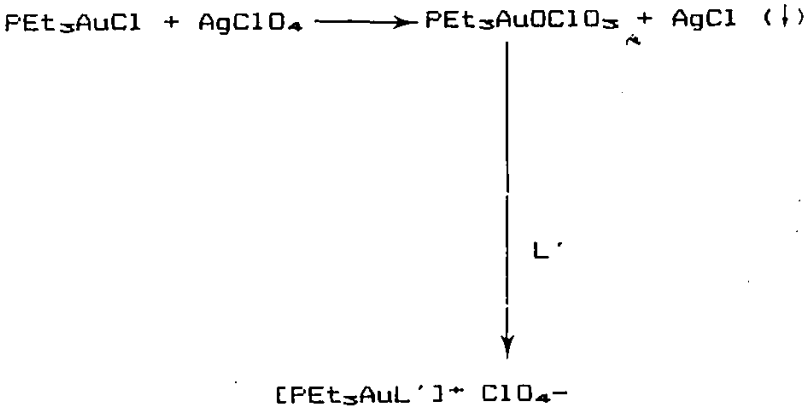
[b]Ligands were prepared as follows: 4, N-acetyl tyrosine ethyl ester plus $NaOCH_3$; 10, cysteine ethylester plus $NaOCH_3$; 14, penicillamine plus 2 NBu_4OH; 15, thioglucose tetraacetate plus $NaOCH_3$; 16 thioglucose plus $NaOCH_3$; 17, thiosuccinic acid plus 3 NBu_4OH; 21, imidazole; 22, histidine methyl ester; 23, methionine methyl ester; 25, no ligand was added.

$$PEt_3AuCl + Na^+L^- \longrightarrow PEt_3AuL + NaCl\ (\downarrow)$$

L = thiolate, phenolate, carboxylate

Figure 2a.

Preparation of PEt_3AuL complexes.



L' = amine, imidazole, thioether

Fig.2b

Preparation of $[PEt_3AuL']^+ ClO_4^-$ complexes.

TABLE 2.2

Solvent Dependence of ^{31}P Nmr Data for PEt_3AuCl

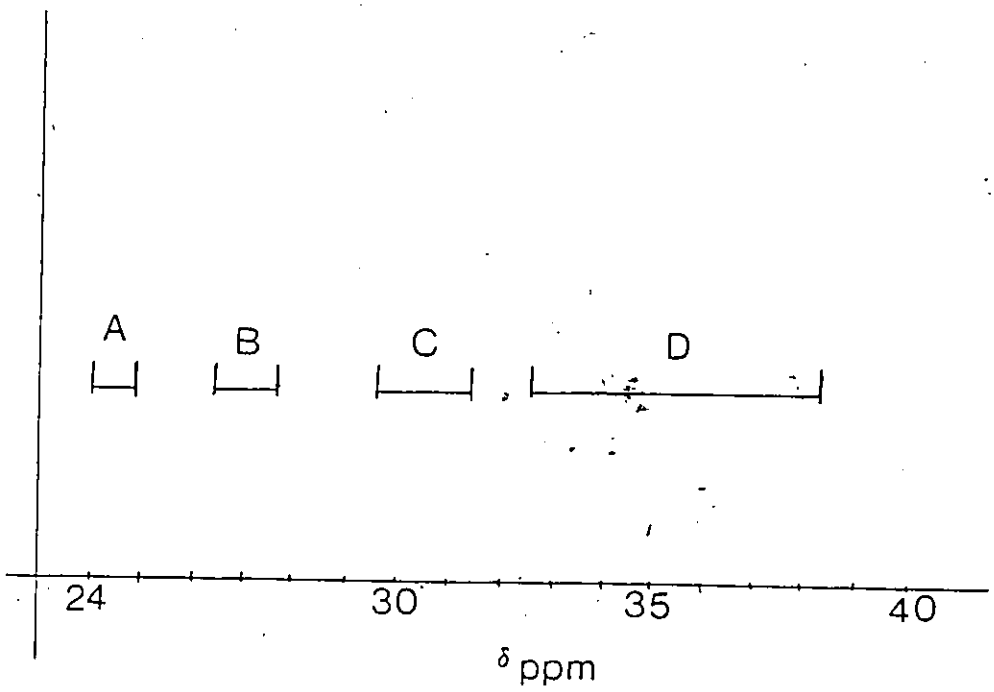
SOLVENT	(ppm)
$CDCl_3$	31.7
$(CD_3)_2CO$	32.7
CD_3OH	33.1
CD_3CN	33.2
$(CD_3)_2SO$	33.7

The ligands employed in the preparation of these compounds were selected so as to mimic possible ligation sites of the 'PEt_3Au' moiety to biomolecules. Thus only the various forms of N, S, and O donor molecules have been utilized. Figure 2.3 shows a graphic representation of the position in the ^{31}P nmr spectra of the various biologically relevant ligands bound to the 'PEt_3Au' moiety. One can clearly see that the thioethers are in the region of 24-25 ppm, the oxygen donor ligands (carboxylate and phenolate) are in the region of 26-28 ppm, the nitrogen donors (amines and imidazoles) are in the region of 29.9-31.9 ppm and the thiolates are in the region of 32-38 ppm.

These chemical shifts on complexation result from the removal of electron density from the P atom upon coordination to the metal. Such effects have been described for other metal-phosphine complexes.[34,39] The ^{31}P nmr chemical shifts of the compounds reported herein are dependent upon the nature of the ligand trans to the PEt_3. [38] The following order is observed:

$$-SR > S^{2-} > Cl^- > NR_3 \sim imidazole > -OR \sim -O_2CR > SR_2$$

This ordering reflects the trans influence of the ligands. Thiolates and sulfide are stronger σ donors than



A thioethers

B carboxylates

C amines, imidazoles

D thiolates

Figure 2.3

(^{31}P nmr scale of biologically relevant ligands)

amines, alkoxides or carboxylates. They are also better π acceptors, facilitating electron density removal from the P atom and thus the observed downfield shift. Presumably the weak donor ability of thioethers prohibits significant metal-ligand π overlap. Thus the chemical shift of the P atom in these complexes appears at high field. The variation of the ^{31}P nmr chemical shift as a function of the solvent is reported for PEt_3AuCl (Table 2.2). Generally, a monotomic relationship is observed between solvent dipole moment and ^{31}P nmr chemical shift. However, the relationship is certainly not linear. the range of variation for the solvents reported herein is 2 ppm. It appears that solvation by polar solvents provides an interaction with the gold complex that results in a deshielding of the P atom. Thus to some extent the chemical shift reflects the polarity of the environment of the 'PEt_3' moiety.

2.3.2 PROTEIN STUDIES

Albumin (HSA or BSA) is treated with mercaptoethanol for a short period of time.[40] This ensures reduction of the one exterior sulfhydryl residue leaving the internal disulfides intact. The reaction of PEt_3AuCl with the albumin was monitored by ^{31}P nmr. The spectra were

obtained after treatment of an aqueous solution of the protein with a methanolic solution of PEt_3AuCl. The spectra showed a sharp signal at 34.2 and 34.9 ppm for HSA and BSA respectively. (See table 2.3, fig 2.4) A weaker broader peak was observed at about 33 ppm. The samples were centrifuged to remove precipitated gold complex. The supernatants gave identical spectra to those described above. The chemical shifts of the singlets were indicative of gold-sulfur binding, based on the comparison to the model compounds. This result is supported by preliminary Mossbauer data mentioned briefly by Brown and Smith.[15] The absence of additional intense resonances implies a considerable specificity of gold for binding to this exterior sulfur residue.

The model compounds exhibited a relationship between solvent polarity and chemical shift. Based on extrapolations to water, the chemical shift observed for the 'PEt_3Au' bound to the protein was upfield of expected values. This upfield shift possibly reflects an environment of lower polarity than the bulk solvent. Thus the sulfhydryl group to which the gold binds is located in a hydrophobic region of the protein.

The SH site of albumin can be blocked by the reaction

TABLE 2.3

protein-PEt_3AuCl Ions: ^{31}P nmr Data*

Protein	(ppm)	
Albumin (HSA)	34.2 (s)	33 (weak, broad)
Albumin (BSA)	34.9 (s)	33 (weak, broad)
SH-blocked (HSA)	33 (broad)	31 (broad)
SH-blocked (BSA)	33 (broad)	31 (broad)

*Spectra recorded in D_2O at T=25°C.

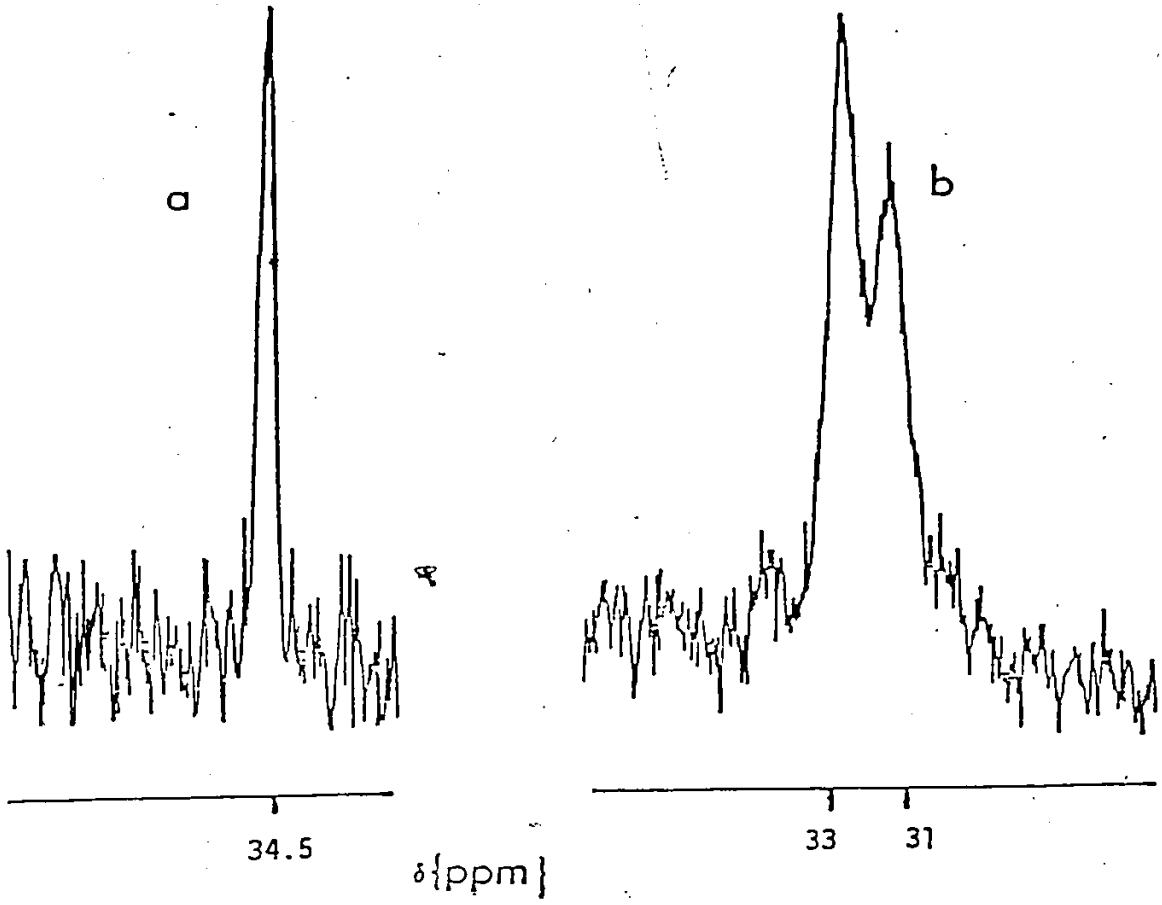


Figure Legend

Figure 2.4 ^{1}H decoupled ^{31}P nmr spectra of

(a) Mercaptalbumin (HSA) after reaction with PEt_3AuCl.

(b) Blocked mercaptalbumin. (HSA), after reaction with PEt_3AuCl

of the albumin with iodoacetamide.[40] Reaction of PEt_3AuCl with blocked proteins was performed in an analogous manner to that described for albumin. Two broad resonances at 33 and 31ppm upfield of the reference were observed in the $^{31}P\{^1H\}$ nmr spectra of the reaction mixtures. These signals indicated clearly the presence of non-specific binding to the protein by PEt_3AuCl. This was also observed to a much lesser extent in the spectra of gold-albumin mixtures. These $^{31}P\{^1H\}$ nmr experiments clearly demonstrate the affinity and specificity of the gold-phosphine complex for the sulfhydryl group in albumin.

Fluorescence properties have been studied to examine biomolecule interactions in a large number of protein systems.[40,41] The fluorescence spectra of albumin were recorded alone and following reaction with PEt_3PAuCl to study the effect of gold binding to the SH residue. Spectra were recorded using an excitation wavelength of 280 nm. No shift in the emission maximum was observed on addition of PEt_3AuCl to the albumin, however, a 12% decrease in signal intensity was seen. (Fig. 2.5). Such a change in intensity could be interpreted as heavy metal quenching of the fluorescence process.[41,42] However, this seems unlikely as the fluorescence of albumin arises from

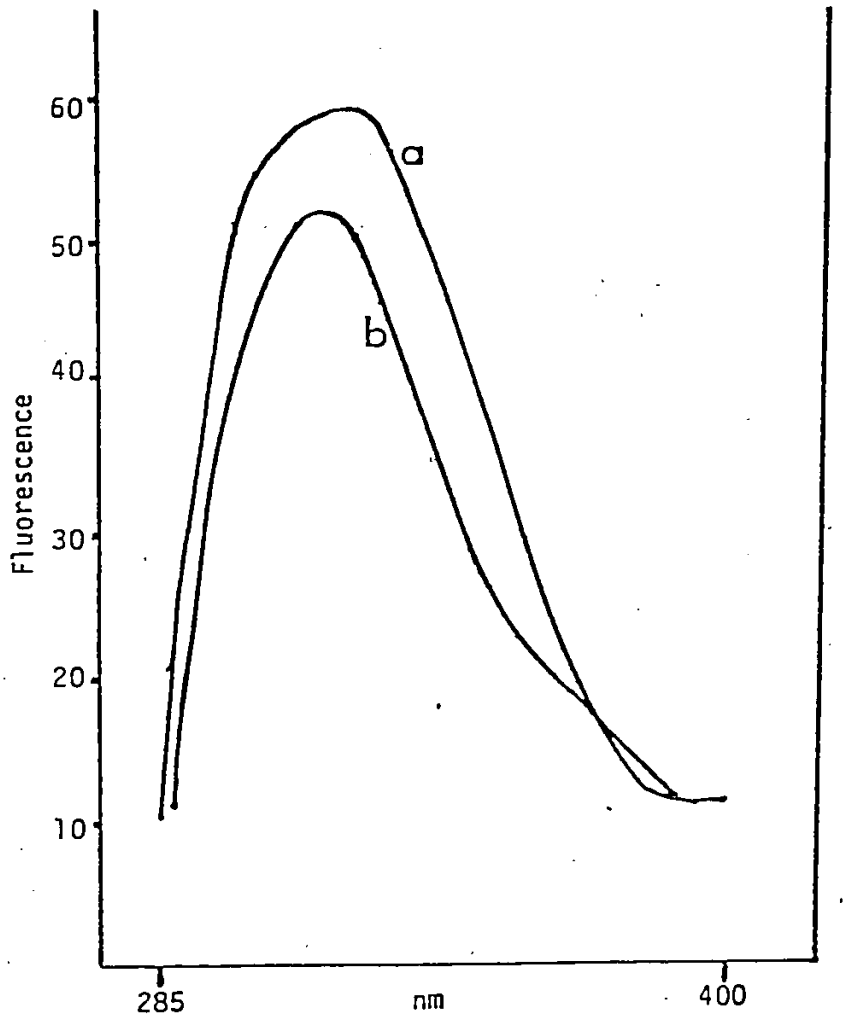


Figure 2.5 Fluorescence spectra of (a) mercaptalbumin (b) mercaptalbumin after reaction with PEt_3AuCl. Protein concentrations in both cases are 2.92 mg/ml.

the tryptophan residue which is a considerable distance from cystein 34, the gold binding site.[43] An alternative interpretation is that gold binding causes a protein conformational change that results in the observed decrease in fluorescence. The degree and nature of such changes are unknown.

In light of these results it is tempting to speculate that the binding of gold to a sulfhydryl residue and an associate protein conformational change are two events leading to drug action. The specific protein system or subsequent steps in such a mechanism are yet to be determined.

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CHAPTER III

Synthesis and Crystal and Molecular Structure of

$MoS_4(AuPEt_3)_2$

A Linear Trinuclear

Heterobimetallic Species

3.1 INTRODUCTION

Recent reviews of metal sulfide species have recognized then to be important as industrial catlaysts, and dehydrosulfurization catalysts.[51,53] In addition a variety of these metal sulfur compounds containing biologically relevant metals have been synthesized and characterized in attempts to model specific enzyme sites, such as ferredoxins.[54,55] Frequently, tetrathiometalates are employed in the synthetic routes to these species. We have shown in chapter 2 that gold-protein interactions involve gold-sulfur bonding. In attempts to characterize this interaction, a bisgold-tetrathiometalate species has been prepared.[44,45,49,52] The preparation, spectroscopic characterization and single crystal X-ray diffraction studies of the linear trinuclear heterobimetallic species $MoS_4(AuPEt_3)_2$ are the subject of this chapter. Although

this species is not a suitable model for gold-albumin binding, the study of this species does provide related structural information.

3.2 Experimental Section

The preparations were done under an atmosphere of dry, oxygen free N_2. Solvents used were reagent grade, distilled from the appropriate drying agents under N_2, and degassed by the freeze-thaw method at least three times prior to use. ^{31}P nmr spectra were recorded on a Bruker CXP 100 spectrometer operating at 36.4 MHz with broad-band decoupling. The ^{31}P chemical shifts are reported relative to 85% H_3PO_4. UV-vis data were recorded employing a Shimadzu 240 spectrometer. Combustion analyses were performed by Guelph Chemical Laboratories, Guelph, Ontario. PEt_3 was purchased from the Strem Chemical Co. Et_3PAuCl was prepared by literature methods employing $HAuCl_4$ received on loan from Johnson-Matthey Ltd.[53]

3.2.1 Preparation of $(NEt_4)_2MoS_4$ (2)

$(NH_4)_2MoS_4$ (1) was prepared by literature methods[57] as follows. Sodium molybdic acid was dissolved in a solution of 30mL of NH_4OH and 10mL of water. Hydrogen sulfide was bubbled rapidly into this solution at room

temperature to saturation. The temperature was raised to 60°C while continuing to bubble in hydrogen sulfide. After 30 minutes, the solution was cooled to 0°C and filtered. The product was washed with isopropanol and ethanol.

Compound (1) was dissolved in a 10% aqueous solution of Et_4NOH. This solution was placed under vacuum for 2 hours to remove the NH_3. The lost water was replaced and the solution filtered into 400mL of isopropanol. The resulting precipitate was isolated by filtration, washed with isopropanol and diethylether and recrystallized from acetonitrile / diethylether. After drying in vacuuo the yield was 68% (based on starting amount of $Na_2MoO_4.2H_2O$)

3.2.2 Preparation of $MoS_4(AuPEt_3)_2$, henceforth known as compound (3). See Figure 3.1

Compound 1 (69mg (0.143mmol) was dissolved in 10mL of ethanol. This solution was added to an ethanolic solution of PEt_3AuCl (100mg, (0.286mmol)). A red crystalline precipitate formed immediately. This product was isolated by filtration and recrystallized from acetonitrile/diethylether to yield 60mg (48%) of compound (3). mpt. 133°C (d). Anal. Calcd for C=16.88; H=3.54. Found: C=17.22; H=3.67. $^{31}P\{^1H\}$ nmr (CH_3CN): , 41.9 ppm

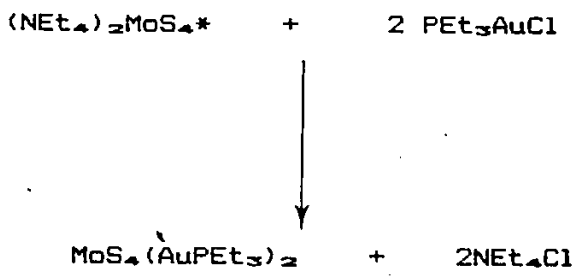

Figure 3.1

Preparation of Complex (3).

* NB. This complex was prepared as outlined in Coord. Chem. Rev., 10, 79, 1973. by E. Dieman and A. Muller.

(s). Uv-vis (CH_3CN): : 490nm (ϵ = 3590 $M^{-1}cm^{-1}$), 430nm (ϵ = 1540 $M^{-1}cm^{-1}$, 375nm (ϵ = 2050 $M_{-1}cm^{-1}$, 345nm (ϵ = 5120 $M^{-1}cm^{-1}$. See Fig. 3.2

3,3 X-ray data Collection and Reduction

Wine-red crystals of (3) were obtained by vapor diffusion of diethylether into a CH_3CN solution of (3). Diffraction experiments were performed on a four-circle Syntex $P2_1$ diffractometer with a graphite-monochromatized MoK radiation. The initial orientation matrix was obtained from 15 machine-centered reflections selected from a rotation photograph. These data were used to determine the crystal system. Partial rotation photographs around each axis were consistent with a monoclinic crystal system. Ultimately, 31 high-angle reflections ($15<2\theta<35°$) were used to obtain the final lattice parameters and the orientation matrix. Machine parameters, crystal data, and data collection parameters are summarized in Table 3.1. The observed extinctions were consistent with the space group $P2_1/C$. $\pm h$, $\pm k$, + l data were collected in one shell ($4.5<2\theta<45.0°$). Three standard reflections were recorded every 197 reflections; their intensities show no statistically significant change over the duration of data collection. The data were proceeded

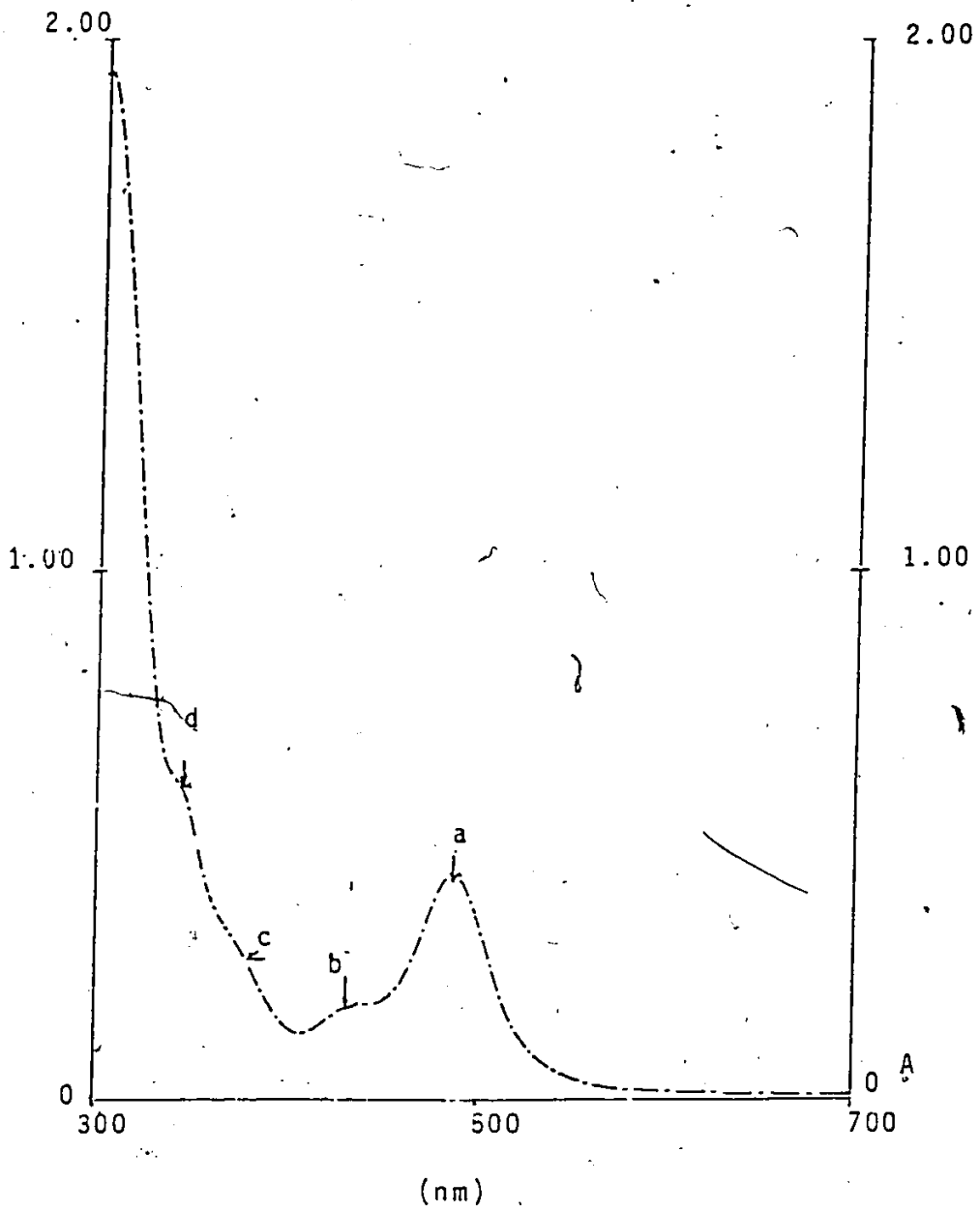

Fig. 3.2

uv-visible spectra of $MoS_4(AuPEt_3)_2$

by using the SHELEX-76 programme package on the computing facilities at the University of Windsor. A total of 2760 reflections with ($F_o < 3\sigma < F_o^2$) were used in the refinement.

3.3.1 Structure Solution and Refinement

Non-hydrogen atomic scattering factors were taken from the tabulation of Cromer and Waber.[57,40] The two Au atom positions were determined by a direct methods technique using the program MULTAN. The remaining non-hydrogen atoms were located from successive difference Fourier map calculations. Full-matrix least-squares refinement, in which all non-hydrogen atoms were assigned isotropic temperature factors gave R = 21.83%. Hydrogen atom contributions for the ethyl hydrogens were included. C-H bond lengths of 0.95 Å were assumed, and hydrogen atom temperature factors were fixed at 1.10 times the isotropic temperature factor of the bonded carbon atom. An empirical absorption correction was applied. In the final cycles of full matrix refinement the Au, Mo, S, P and methylene carbon atoms were assigned anisotropic temperature factors, the methyl groups were refined as rigid rotors with isotropic thermal parameters, and all hydrogen atom contributions were included but not refined. This gave R = 4.88% and Rw = 5.25%. The maximum

Table 3.1 Summary of Crystal Data, Intensity Collection and Structure Solution

formula	$MoS_4(AuPEt_3)_2$
cryst color, form	wine red, blocks
a, Å	18.370 (3)
b, Å	7.928 (1)
c, Å	18.733 (4)
β, deg	120.87 (1)
cryst syst	monoclinic
space group	$P2_1/c$
V, Å^3	2341.7 (6)
d calcd, gcm^{-3}	2.42
Z	4
cryst dimens, mm	0.31 x 0.12 x 0.25
abs. coeff (μ), cm^{-1}	129.72
radiation (λ, Å)	Mo Kα (0.71069) (graphite monochromator)
temp, °C	24
scan speed, deg/min	2.0-5.0° (θ/2θ scan)
scan range, deg	2.0° below $K\alpha_1$, 1.0° above $K\alpha_2$
bkgd/scan time ratio	0.5
data collecd	,2θ of 4.5-50.0° (±h,±k,+l)
total no. of data collecd	6230
no. of unique data ($Fo^2 \geq 3\ Fo^2$)	2760
no. of variables	189
R, %	4.88
Rw, %	5.25

Δ/σ on any of the parameters excluding those associated with the methyl groups in the final cycles was 0.051. The shift on the parameters of the methyl groups was larger reflecting the disordered nature of these CH_3 moieties. Since no information regarding the partial occupancy positions of the methyl hydrogen atoms could be derived from difference map calculations no attempt was made to model their disorder. The final difference Fourier map calculation showed no peaks of chemical significance; the largest peak was 1.0 electrons and was associated with the Au1-S1 moiety. The following data are tabulated: positional parameters (Table 3.2); interatomic distances and angles (Table 3.3). Temperature factors (Table S-I), Hydrogen atom parameters (Table S-II) angles and distances associated with the PEt_3 groups. (Table S-III) and values of 10 |Fo| and 10 |Fc| (Table S-IV) have been placed in Appendix A.

RESULTS AND DISCUSSION

The reaction of Et_3PAuCl and $(NEt_4)_2MoS_4$ was performed in ethanol according to Figure 3.1 Upon reaction, a microcrystalline orange-red precipitate was formed immediately. This product was recrystallized from

Table 3.2 Positional Parameters *

atom	x	y	z
Au1	1673(1)	3080(1)	5804(1)
Au2	3200(1)	4509(1)	3918(1)
Mo	2477(1)	3681(2)	4887(1)
S1	1229(3)	2418(6)	4379(2)
S2	3050(3)	4273(6)	6233(3)
S3	3322(3)	1986(6)	4702(3)
S4	2278(3)	6110(6)	4226(3)
P1	948(3)	2784(5)	6481(2)
P2	3830(3)	5196(6)	3178(3)
C1	1389(12)	4081(23)	7419(9)
C2	1376(13)	5986(26)	7235(13)
C3	916(11)	645(22)	6836(10)
C4	395(11)	412(22)	7224(10)
C5	-163(9)	3446(22)	5823(9)
C6	-670(12)	2274(22)	5062(12)
C7	4575(13)	3497(32)	3228(14)
C8	5181(15)	2905(33)	4112(14)
C9	4448(14)	7150(33)	3533(14)
C10	4006(20)	8656(36)	3563(19)
C11	3086(11)	5517(24)	2078(11)
C12	3466(15)	5878(30)	1532(14)

* Parameters given X 10^4

Table 3.3 Selected Bond Distances and Angles *

Mo-S1	2.258(4)	Mo-S2	2.182(4)
Mo-S3	2.234(4)	Mo-S4	2.205(5)
Au1-S1	2.362(4)	Au1-S2	2.466(5)
Au2-S3	2.409(5)	Au2-S4	2.432(4)
Au1-P1	2.277(4)	Au2-P2	2.275(4)
Au1-Mo	2.815(1)	Au2-Mo	2.814(1)
S1-Mo-S2	111.5(2)	S1-Mo-S3	109.5(2)
S1-Mo-S4	108.3(2)	S2-Mo-S3	109.1(2)
S2-Mo-S4	106.4(2)	S3-Mo-S4	112.0(2)
S1-Au1-S2	99.0(1)	S3-Au2-S4	99.0(1)
S1-Au1-P1	127.8(1)	S2-Au1-P1	133.1(1)
S3-Au2-P2	129.6(2)	S4-Au2-P2	131.4(2)
Au1-S1-Mo	75.0(1)	Au1-S2-Mo	74.3(1)
Au2-S3-Mo	74.5(1)	Au2-S2-Mo	74.5(1)

* Bond lengths in Å, angles in degrees.

acetonitrile/diethylether and yielded wine red crystals; which were suitable for X-ray analysis. $^{31}P\{^1H\}$ nmr spectroscopy of this material, showed a singlet at 41.9 ppm. Comparison of this chemical shift with Table 2.1 in Chapter II, suggests that gold sulfur binding occurs. The singlet is consistent with equivalent uncoupled phosphorus atoms.

UV-visible data of compound (3) showed absorptions at 490, 430, 375, 345 nm. See Figure 3.2. These spectral feature are reminiscent of the spectrum of $(NCH_4)_2[NCCu(MoS_4)]$.[52] (See Fig. 3.3) In trimetallic complexes, with tetrahedral coordination of the central atom, the coordination geometry gives rise to considerable disturbances of the electronic spectra. The bridging MoS_4 moiety results in a decrease in intensity of the bands at 490nm and 275nm, while the band at 345nm is practically unaltered. This type of pattern is also exhibited by the spectra of $MoS_4(AuPEt_3)_2$ which would tend to suggest the the presence of a bridging tetrahedral MoS_4 moiety.

C,H analysis confirmed the formulation of the product to be $MoS_4(AuPEt_3)_2$. Formation of compound 3 takes place regardless of the Au:Mo ratio. Spectroscopic experiments where the Au:Mo ratio was varied between 2:1 and 1:1

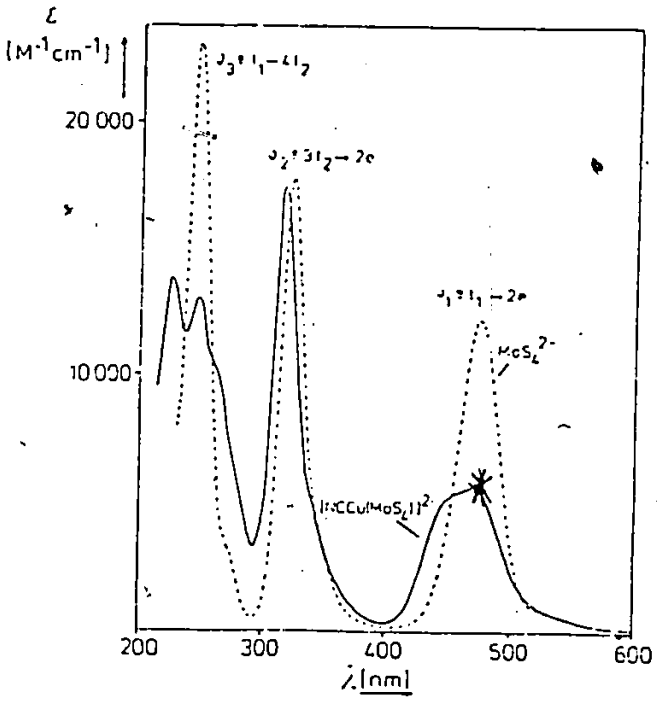

Figure 3.3

Electronic spectra of $(NMe_4)_2[NCCu(MoS_4)]$ and $(NMe_4)_2MoS_4$ in acetonitrile.

demonstrated there was no other isolated species. This result is consistent with the known affinity of gold for sulfur ligands [41] as well as the propensity of the MoS_4^{2-} to act as a tetradentate bridging unit.

Single crystals of compound (3) were obtained by vapor diffusion of diethyl ether into an acetonitrile solution of complex (3). X-ray crystallographic data revealed that the crystals are made of unit cells each containing 4 discrete molecules. The closest non-bonded contact between molecules was 2.852 Å (H11 B-Au1). Selected interatomic distances and angles are tabulated in Table 3.2. An ORTEP drawing of the molecule is shown in Figure 3.4. The MS_4^{2-} acts as a doubly bridging ligand and the M'atoms each have one PEt_3 ligand. In this case the coordination number of gold is three. This results in one atom exhibiting a pseudo tetrahedral environment while the other exhibits a pseudotrigonal environment. Complex (3) has 4 S atoms about the Mo atom with average Mo-S bond distances of 2.220(9) Å . There are 2 chemically equivalent Au atoms and 2 bridging sulfur atoms and a phosphorus atom of the PEt_3 moiety. The P-Au-Mo-Au-P fragment is observed to be essentially linear. The Au-S and Au-P bond lengths are similar to those which are observed in other complexes.

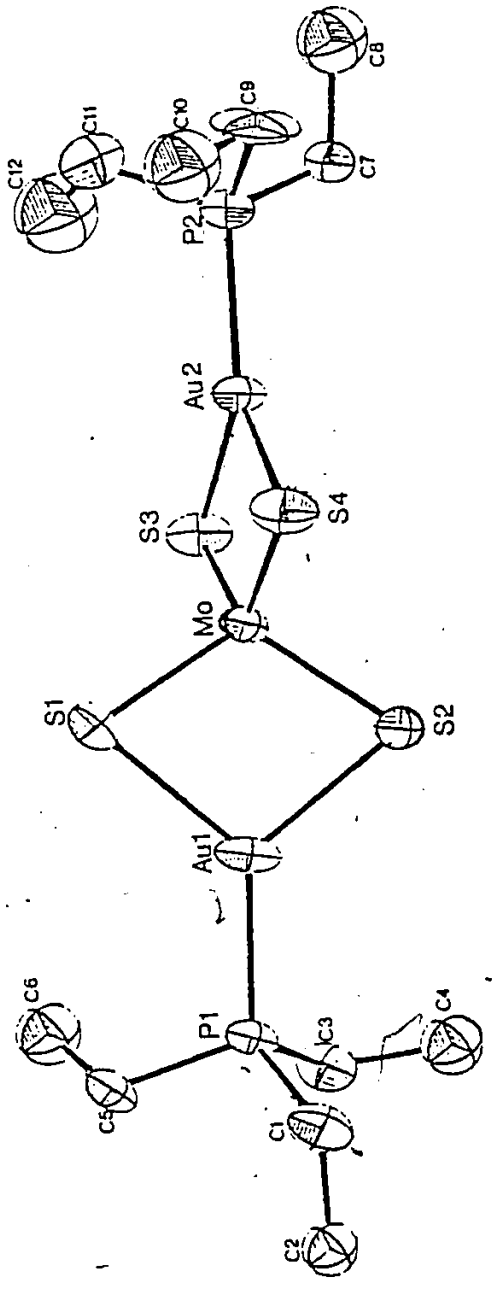

Figure3.4 ORTEP Drawing of the molecule, $MoS_4(AuPEt_3)_2$

50% thermal ellipsoids are shown. Hydrogen atoms are omitted for clarity.

The structure of $MoS_4(AuPEt_3)_2$ consist of a tetrahedral MoS_4 core with two chemically equivalent PEt_3Au moieties. The average gold-sulfur bond length is 2.418 (5) Å and the average gold-phosphorus bond length is 2.277 Å. The Au-Mo bond distance is 2.85 Å which is 0.04 Å longer than the sum of the atomic radii for Au-Mo. This would suggest a metal-metal interaction. (See Fig. 3.5)

A tungsten analogue of complex (3) is $[P(Ph_2PCH_3Au]_2WS_4$.[48b] (See Fig. 3.6) This complex consists of a WS_4 core. It has S-W-S angles ranging from 108.54 to 111.55 ° with an average tungsten-sulfur bond length of is 2.22 Å. The average gold-sulfur bond length is 2.424 A and the average gold-phosphorus bond length is 2.27 Å. The Au-W distance is 2.84 Å. The sum of the atomic radii is 0.06 Å longer than the 2.84 Å Au-W distance.

The gold-tungsten and the gold-molybdenum complexes exhibit similar gold-phosphorus bond lengths. There appears to be no significant difference in the gold-phosophorus bond length between the two complexes. The average gold-phosphorus bond lengths are 2.27 and 2.26 Å respectively. The gold-sulfur bond lengths in both the gold-tungsten and gold-molybdenum complexes are not significantly different. The average gold-sulfur bond

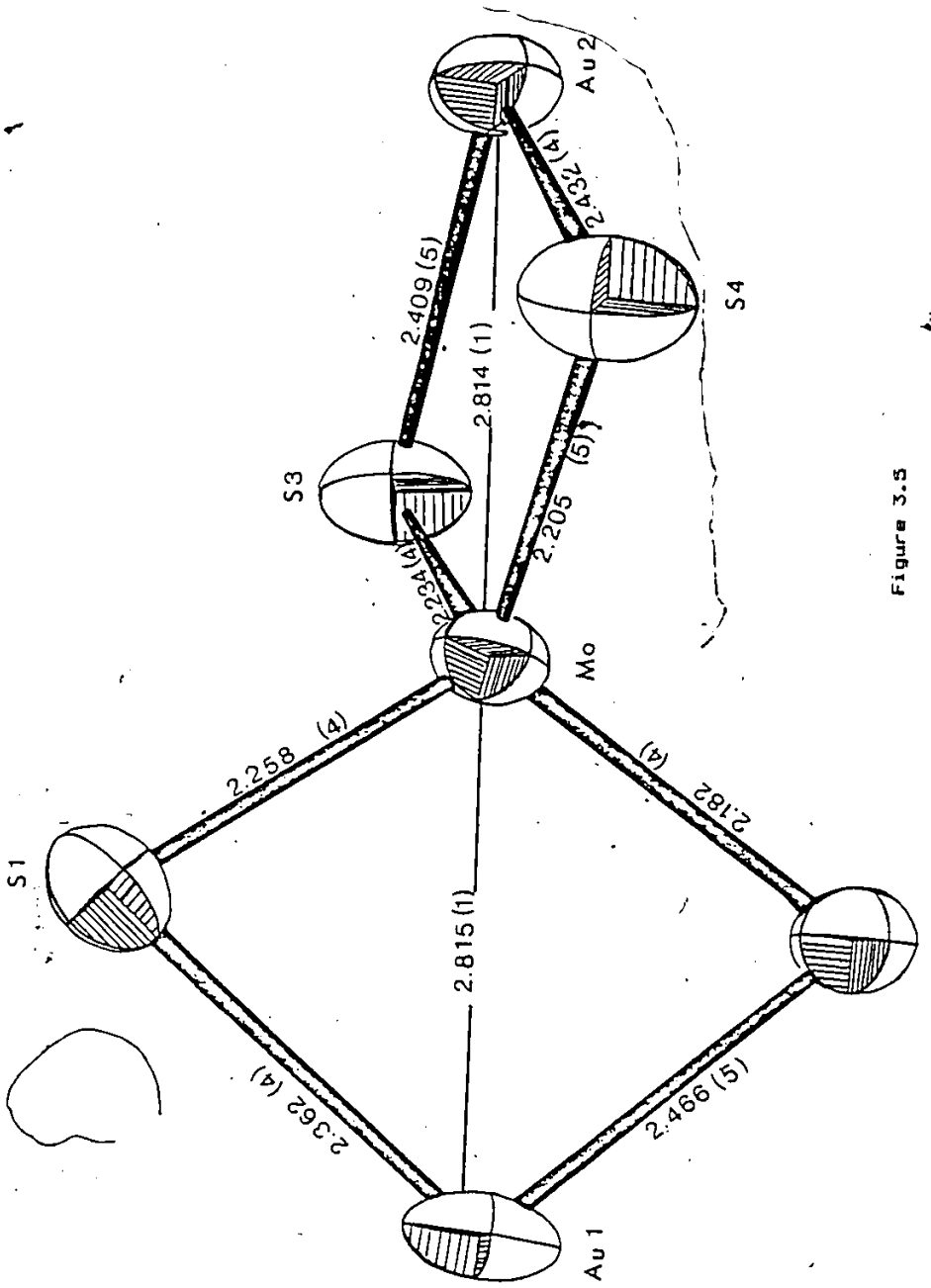

Figure 3.5 ORTEP of the core of $MoS_4(AuPEt_3)_2$

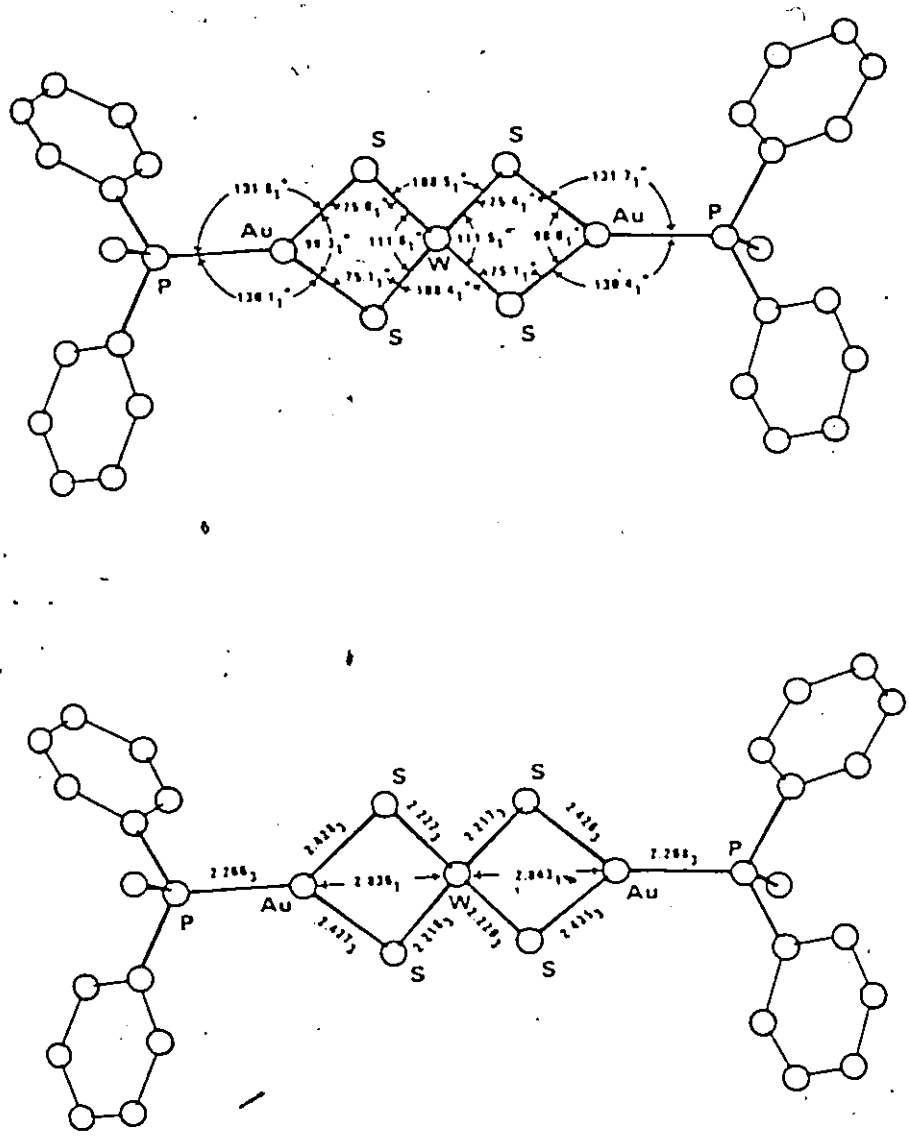

Figure 3.6

ORTEP drawing of the tungsten analogue of complex (3)

lengths for both complexes are 2.219 Å. The gold-metal bond distances are 2.814 and 2.815 Å apart. These show no significant difference between the two different metals. Molecular orbital calculations are described for compounds containing an $M'S_2MS_2M'$ unit. These studies also show that delocalized molecular orbitals occur over the M-M' moiety. Such interactions are thought to be the logical consequence of putting two metals of diverse oxidation states in close proximity. However, it is also clear that on formation of the metal-metal bond the formal oxidation states of the metal centers is altered.

Although complex (3) is not a suitable model for Au bonding to CYS-34 in serum albumin pertinent structural data regarding gold-sulfur interactions is derived. The structure of complex (3) shows a bond angle of 127.8°, and Au-S and Au-P bond lengths of 2.42 and 2.29 Å respectively. While the stuctural data of Auranofin [13] shows a nearly linear S-Au-P bond angle of 173.6° and Au-S and Au-P bond lengths respectively. The difference in S-Au-P is consistent with the change in coordination geometry of the gold atom from linear to trigonal planar. The Au-P bond lengths agree closely, whereas the Au-S bond length in the two structures are found to be significantly different. The difference between the two structures

clearly arises as a result of the differing nature of the two ligands involved in complex (3) and Auranofin; that is thiomolybdate versus thiolate.

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CHAPTER IV

SUMMARY

In this thesis it is shown that PEt_3AuCl binds to the albumin at the cystein (SH) 34 position. Upon blocking of this binding site with iodoacetamide, the specific binding is eliminated. This is demonstrated by the non-specific binding of the gold to the protein which is observed by $^{31}P\{^1H\}$ nmr spectroscopy. Fluorescence studies suggest that there is a conformational change of the protein upon binding of the drug. This combination of the drug binding to albumin and the conformational change associated with this interaction could possibly lead to drug action. However, whether albumin acts as a scavenger to eliminate the drug or whether it acts as a medium for the transport of gold to the target site is yet to be determined. The possibility that albumin tranfers the gold drugs to red blood cells and that the blood transports the gold must not be overlooked.

Nonetheless, we have clarified the nature and results of gold-albumin binding. This may well be an important step in beginning to understand the mechanism of action of these anti-arthritic drugs.

In the second half of this thesis, a trinuclear heterobimetallic species of $MoS_4(AuPEt_3)_2$ is examined by $^{31}P\{^1H\}$ nmr, uv-vis spectroscopy and single crystal X-ray diffraction studies. Although this complex is not directly related to the gold-cysteine 34 bond in albumin, it does have some similarities, and does provide pertinent structural data with respect to gold-sulfur bonding. The work described in this thesis does contribute to the general information required for the understanding of the anti-arthritic action of gold drugs. However, there remain many unanswered questions. Further studies of the structural chemistry of gold in biological systems requires an interdisciplinary approach in which inorganic and biochemists contribute their respective expertise.

APPENDIX A

This Appendix provides a listing of the structure factors (observed vs. calculated for $MoS_4(AuPEt_3)_2$.

Table S-I Temperature Factors *

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Mo	30.5(7)	41.5(8)	24.9(6)	1.3(5)	17.2(5)	-1.3(6)
Au1	50.6(4)	42.3(4)	41.3(4)	11.1(3)	36.0(3)	9.9(3)
Au2	40.2(4)	72.9(5)	34.7(4)	-5.7(3)	26.8(3)	-14.3(4)
S1	36(2)	60(3)	60(2)	-5(2)	20(2)	-9(2)
S2	53(3)	72(3)	29(2)	-7(2)	21(2)	-16(2)
S3	45(3)	56(3)	51(2)	6(2)	33(2)	9(2)
S4	50(3)	51(3)	53(3)	12(2)	36(2)	7(2)
P1	42(2)	29(2)	29(2)	4(2)	25(2)	5(2)
P2	50(3)	59(3)	44(2)	-12(2)	35(2)	-17(2)
C1	56(12)	77(13)	26(8)	-7(8)	25(8)	-13(10)
C2	62(5)					
C3	58(11)	54(11)	32(8)	11(7)	25(8)	7(9)
C4	45(4)					
C5	29(9)	58(11)	38(8)	7(8)	17(7)	15(8)
C6	61(5)					
C7	59(13)	127(21)	87(15)	-8(14)	67(13)	11(14)
C8	79(7)					
C9	67(15)	110(20)	69(14)	-13(13)	40(12)	-30(15)
C10	114(10)					
C11	60(12)	62(12)	61(11)	-26(9)	48(10)	-25(10)
C12	74(6)					

* multiplied by 10^3
the form of the thermal ellipsoid is
$\exp[-2\pi^2(U_{11}a^{*2}h^2 + U_{22}b^{*2}k^2 + U_{33}c^{*2}l^2 + 2U_{12}a^*b^*hk + 2U_{13}a^*c^*hl + 2U_{23}b^*c^*kl)]$, where only one thermal parameter is given it corresponds to the isotropic U value.

Table S-II Hydrogen Atom Parameters *

atom	x	y	z	U
H1A	195(1)	375(2)	778.3(9)	0.0570
H1B	109(1)	390(2)	769.6(9)	0.0570
H2A	155(1)	620(3)	781(1)	0.0704
H2B	101(1)	685(3)	689(1)	0.0704
H2C	185(1)	596(3)	717(1)	0.0704
H3A	147(1)	32(2)	724(1)	0.0643
H3B	71(1)	-8(2)	636(1)	0.0643
H4A	44(1)	-77(2)	733(1)	0.0488
H4B	-15(1)	67(2)	678(1)	0.0488
H4C	51(1)	99(2)	773(1)	0.0488
H5A	-18.3(9)	456(2)	562.0(9)	0.0618
H5B	-40.5(9)	344(2)	616.9(9)	0.0618
H6A	-117(1)	290(3)	474(1)	0.0663
H6B	-76(1)	141(3)	537(1)	0.0663
H6C	-51(1)	179(3)	469(1)	0.0663
H7A	488(1)	394(3)	299(1)	0.0691
H7B	426(1)	256(3)	290(1)	0.0691
H8A	561(2)	214(3)	422(1)	0.0820
H8B	481(2)	239(3)	425(1)	0.0820
H8C	541(2)	389(3)	445(1)	0.0820
H9A	465(1)	737(3)	316(1)	0.0766
H9B	491(1)	696(3)	409(1)	0.0766
H10A	454(2)	902(4)	399(2)	0.1208
H10B	363(2)	875(4)	377(2)	0.1208
H10C	381(2)	934(4)	307(2)	0.1208
H11A	275(1)	645(2)	203(1)	0.0682
H11B	275(1)	455(2)	187(1)	0.0682
H12A	302(2)	611(3)	97(1)	0.0804
H12B	377(2)	492(3)	152(1)	0.0804
H12C	383(2)	682(3)	174(1)	0.0804

* Positional parameters are multiplied by 10^3.

Table S-III Bond Distances and Angles Associated with the PEt$_3$ Groups *

P1-C1	1.80(2)	P1-C3	1.83(2)
P1-C5	1.87(2)	P2-C7	1.91(2)
P2-C9	1.84(2)	P2-C11	1.79(2)
C1-C2	1.55(3)	C3-C4	1.49(2)
C5-C6	1.54(2)	C7-C8	1.50(3)
C9-C10	1.47(3)	C11-C12	1.53(2)
Au1-P1-C1	110.7(6)	Au1-P1-C3	115.4(6)
Au1-P1-C5	113.4(5)	Au2-P2-C7	113.1(7)
Au2-P2-C9	114.5(7)	Au2-P2-C11	111.5(6)
P1-C1-C2	114(1)	P1-C3-C4	116(1)
P1-C5-C6	112(1)	P2-C7-C8	114(2)
P2-C9-C10	116(2)	P2-C11-C12	114(1)
C1-P1-C3	105.7(8)	C1-P1-C5	105.7(8)
C3-P1-C5	105.2(8)	C7-P2-C9	105(1)
C7-P2-C11	106.3(9)	C9-P2-C11	105(1)

* Bond lengths in Å, angles in degrees.

Table SIV: |Fo|vs|Fc| for $MoS_4(AuPEt_3)_2$. E.M. Kinsch, D.W. Stephan

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MDAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	0	0	3800	-4430	1	2	0	2325	-2301	2	4	0	884	920	4	8	0	842	865	13	1	1	1308	1342
3	0	0	524	492	2	2	0	943	-973	3	4	0	443	375	6	8	0	1370	-1363	14	1	1	1123	-1194
4	0	0	1608	-1736	3	2	0	4177	4552	4	4	0	1761	-1772	8	8	0	1030	982	15	1	1	847	-819
5	0	0	1797	-1830	4	2	0	818	804	5	4	0	907	-926	2	9	0	400	358	16	1	1	1073	1052
6	0	0	3165	3211	5	2	0	1493	-1432	6	4	0	1080	1065	4	9	0	418	-448	18	1	1	462	-436
7	0	0	866	840	6	2	0	288	114	7	4	0	414	355	5	9	0	750	-697	-15	2	1	630	-445
8	0	0	1641	-1649	7	2	0	443	-466	8	4	0	907	-918	-16	1	1	515	383	-14	2	1	458	271
9	0	0	504	479	8	2	0	857	-834	10	4	0	897	874	-17	1	1	587	-425	-13	2	1	1415	1435
10	0	0	695	-697	9	2	0	1304	1305	12	4	0	814	-803	-16	1	1	361	230	-12	2	1	518	-387
11	0	0	1426	-1260	10	2	0	1144	1141	1	5	0	862	-811	-14	1	1	1413	-1467	-11	2	1	1981	-2057
12	0	0	2360	2320	11	2	0	1605	-1647	2	5	0	857	733	-13	1	1	814	549	-10	2	1	430	-267
13	0	0	1494	1544	12	2	0	705	-717	3	5	0	954	-947	-12	1	1	1899	1922	-9	2	1	691	733
14	0	0	2324	-2314	13	2	0	535	558	4	5	0	1792	-1797	-11	1	1	1168	-1225	-8	2	1	565	557
15	0	0	732	-677	14	2	0	427	-353	5	5	0	952	915	-10	1	1	1173	-1179	-7	2	1	674	762
16	0	0	606	769	15	2	0	553	510	6	5	0	987	964	-9	1	1	998	1032	-6	2	1	739	-815
17	0	0	329	-309	16	2	0	731	744	7	5	0	970	-967	-7	1	1	340	-397	-5	2	1	1597	-1668
18	0	0	436	258	17	2	0	730	-707	8	5	0	534	522	-6	1	1	1267	1237	-3	2	1	240	30
1	1	0	4129	-4016	18	2	0	437	-399	9	5	0	950	943	-5	1	1	827	-838	-2	2	1	740	732
2	1	0	2248	-2301	1	3	0	529	-475	10	5	0	1253	-1233	-4	1	1	1334	-1454	-1	2	1	2024	2495
3	1	0	691	622	2	3	0	2432	2466	11	5	0	559	-561	-3	1	1	1021	990	0	2	1	473	-461
4	1	0	501	410	3	3	0	1262	1249	12	5	0	586	562	-2	1	1	799	-678	1	2	1	4089	-4083
5	1	0	2520	2545	4	3	0	2355	-2350	0	6	0	254	196	-1	1	1	2105	-2013	2	2	1	371	305
6	1	0	1214	1175	5	3	0	964	-947	3	6	0	761	-732	0	1	1	4400	4536	3	2	1	2516	2229
7	1	0	1387	-1420	6	3	0	698	659	5	6	0	1004	948	1	1	1	937	924	6	2	1	326	350
8	1	0	708	-704	7	3	0	795	-807	9	6	0	1002	-959	2	1	1	5003	-5106	7	2	1	1205	-1116
10	1	0	332	343	8	3	0	775	715	11	6	0	566	519	3	1	1	875	810	8	2	1	780	-742
11	1	0	1639	1592	9	3	0	618	588	1	7	0	1086	-1024	4	1	1	2597	2518	9	2	1	822	780
13	1	0	1855	-1828	10	3	0	1251	-1167	5	7	0	861	856	5	1	1	909	-818	10	2	1	332	356
14	1	0	579	-544	12	3	0	824	767	7	7	0	1336	-1349	6	1	1	1298	1307	11	2	1	670	668
15	1	0	787	902	13	3	0	637	-586	8	7	0	542	-547	7	1	1	1021	1006	12	2	1	391	365
16	1	0	436	347	15	3	0	878	861	9	7	0	630	575	8	1	1	1641	-1589	13	2	1	1511	-1468
17	1	0	303	195	16	3	0	574	-543	11	7	0	374	345	10	1	1	851	795	14	2	1	719	-650
18	1	0	359	140	17	3	0	717	-634	12	7	0	364	255	11	1	1	828	-797	15	2	1	1364	1316
0	2	0	492	-493	0	4	0	223	190	0	8	0	746	-680	12	1	1	400	339	16	2	1	436	394

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR HOAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
17	2	1	585	-548	-16	4	1	847	818	-9	5	1	1305	-1300	3	6	1	647	603	5	8	1	398	-437
-18	3	1	511	215	-15	4	1	554	-503	-8	5	1	756	-750	4	6	1	553	499	-6	9	1	532	-581
-17	3	1	619	-533	-14	4	1	1365	-1348	-7	5	1	1154	1158	5	6	1	866	826	-4	9	1	476	436
-16	3	1	486	299	-12	4	1	413	287	-6	5	1	1259	1323	6	6	1	304	414	-1	9	1	367	213
-15	3	1	1269	1281	-11	4	1	583	533	-4	5	1	1530	-1577	7	6	1	1554	-1517	-18	0	2	751	604
-13	3	1	794	-841	-10	4	1	1743	1810	-3	5	1	1468	-1530	8	6	1	712	-699	-16	0	2	952	-959
-12	3	1	672	-686	-9	4	1	556	-554	-2	5	1	1070	1056	9	6	1	1421	1361	-15	0	2	919	896
-11	3	1	947	-961	-8	4	1	2218	-2402	-1	5	1	1329	1312	10	6	1	388	302	-13	0	2	455	274
-10	3	1	1382	1467	-7	4	1	720	755	0	5	1	722	676	11	6	1	581	-497	-12	0	2	3182	3380
-9	3	1	1933	1983	-6	4	1	579	514	2	5	1	791	-766	12	6	1	331	233	-11	0	2	1208	-1214
-8	3	1	1155	-1302	-5	4	1	298	-204	3	5	1	1408	-1352	-9	7	1	480	148	-10	0	2	4349	-4808
-7	3	1	1351	-1453	-4	4	1	2037	2140	4	5	1	521	-551	-8	7	1	502	432	-8	0	2	2149	2199
-6	3	1	384	357	-3	4	1	467	-487	5	5	1	961	886	-6	7	1	817	-855	-6	0	2	1195	1239
-5	3	1	368	-186	-2	4	1	3506	-3571	6	5	1	1136	1088	-5	7	1	472	234	-5	0	2	776	-759
-4	3	1	828	-845	-1	4	1	268	299	7	5	1	346	369	-4	7	1	933	1010	-4	0	2	2144	-1993
-3	3	1	2745	2821	0	4	1	1918	1868	8	5	1	715	-657	-3	7	1	369	137	-3	0	2	283	-253
-2	3	1	645	604	1	4	1	726	749	9	5	1	1228	-1224	-2	7	1	486	-505	-2	0	2	1887	1920
-1	3	1	3595	-3593	2	4	1	2305	2150	10	5	1	438	447	-1	7	1	462	-413	0	0	2	3234	3172
0	3	1	1757	-1569	4	4	1	3470	-3355	11	5	1	1255	1177	0	7	1	311	-303	2	0	2	5808	-7234
1	3	1	337	323	5	4	1	419	-406	15	5	1	684	-684	2	7	1	468	369	3	0	2	1483	-1307
2	3	1	1063	1028	6	4	1	2068	2005	-14	6	1	531	447	3	7	1	470	436	4	0	2	3825	3722
3	3	1	3578	3393	7	4	1	420	-191	-13	6	1	429	295	4	7	1	377	428	5	0	2	688	630
4	3	1	532	-479	8	4	1	312	305	-12	6	1	432	-459	6	7	1	962	-935	6	0	2	338	305
5	3	1	3064	-3028	9	4	1	622	603	-11	6	1	787	-674	7	7	1	462	-405	7	0	2	297	242
7	3	1	677	641	10	4	1	1885	-1841	-9	6	1	369	-271	8	7	1	975	888	8	0	2	1777	-1729
8	3	1	310	-324	11	4	1	739	-744	-7	6	1	1243	1283	10	7	1	526	-438	10	0	2	1644	1583
9	3	1	756	675	12	4	1	1498	1416	-6	6	1	462	-281	-10	8	1	534	314	12	0	2	407	335
10	3	1	932	920	13	4	1	435	286	-5	6	1	1692	-1751	-8	8	1	407	-366	14	0	2	1435	-1390
11	3	1	1250	-1211	15	4	1	369	253	-3	6	1	1126	1189	-4	8	1	426	98	15	0	2	1099	-1097
12	3	1	1342	-1315	16	4	1	787	-734	-2	6	1	504	457	-3	8	1	380	-329	16	0	2	1450	1363
13	3	1	568	550	-16	5	1	624	-535	-1	6	1	445	459	-2	8	1	457	-472	17	0	2	827	782
14	3	1	720	640	-15	5	1	562	-465	0	6	1	292	-237	-1	8	1	303	306	-17	1	2	386	-348
15	3	1	402	354	-13	5	1	817	789	1	6	1	1240	-1239	0	8	1	434	381	-16	1	2	524	402
17	3	1	615	-577	-12	5	1	533	520	2	6	1	678	-635	3	8	1	402	395	-15	1	2	657	602

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MOAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-13	1	2	517	453	-9	2	2	1881	1977	-2	3	2	2130	-2057	7	5	2	331	-255	-14	1	3	447	398
-12	1	2	917	-972	-8	2	2	535	-568	0	3	2	1927	1807	8	5	2	740	-741	-13	1	3	1068	-1097
-11	1	2	2486	-2621	-7	2	2	2401	-2439	1	3	2	293	-292	9	5	2	732	779	-12	1	3	1499	1591
-10	1	2	394	108	-6	2	2	617	-619	3	3	2	1256	1170	11	5	2	1093	-1081	-11	1	3	665	689
-9	1	2	2443	2631	-5	2	2	1709	1720	4	3	2	1619	-1512	12	5	2	539	509	-10	1	3	3183	-3388
-8	1	2	1319	1353	-4	2	2	880	844	5	3	2	1638	-1567	13	5	2	463	448	-8	1	3	2908	3054
-6	1	2	1042	-1026	-3	2	2	1330	1237	6	3	2	1440	1405	14	5	2	493	-482	-7	1	3	901	-871
-5	1	2	3274	-3390	-2	2	2	319	299	7	3	2	726	704	0	6	2	403	391	-6	1	3	1130	-1024
-3	1	2	1578	1580	-1	2	2	3814	-3761	8	3	2	939	-909	1	6	2	310	-329	-5	1	3	1389	1337
-2	1	2	1060	963	0	2	2	2347	-2277	10	3	2	321	-307	2	6	2	835	-821	-4	1	3	992	-906
-1	1	2	575	792	1	2	2	1765	1674	12	3	2	1234	1156	4	6	2	690	668	-3	1	3	253	-213
0	1	2	1930	-1827	2	2	2	292	259	14	3	2	714	-705	5	6	2	768	715	-2	1	3	1787	1795
1	1	2	3929	-4528	3	2	2	737	657	15	3	2	319	317	7	6	2	537	-427	-1	1	3	368	-328
2	1	2	194	-40	4	2	2	1222	1133	-7	4	2	308	-63	9	6	2	377	-265	0	1	3	746	-746
3	1	2	3498	3352	5	2	2	2419	-2257	-6	4	2	1602	1687	11	6	2	607	586	1	1	3	2234	2294
4	1	2	1441	1300	6	2	2	970	-903	-4	4	2	1935	-1924	1	7	2	850	-900	2	1	3	1750	-1715
5	1	2	457	-455	7	2	2	1595	1508	-3	4	2	355	-329	3	7	2	1094	1018	3	1	3	2003	-2073
6	1	2	336	-242	9	2	2	306	-223	-2	4	2	1660	1688	4	7	2	550	536	4	1	3	3045	3044
7	1	2	911	-838	10	2	2	409	343	0	4	2	528	-543	7	7	2	773	-759	5	1	3	645	623
9	1	2	904	849	11	2	2	756	-733	1	4	2	479	456	8	7	2	450	-323	6	1	3	2099	-2011
10	1	2	366	261	12	2	2	930	-912	3	4	2	268	-229	9	7	2	1039	1027	7	1	3	402	368
12	1	2	641	-553	13	2	2	1191	1177	4	4	2	592	-574	10	7	2	573	518	9	1	3	723	-690
13	1	2	1300	-1274	14	2	2	836	826	5	4	2	1094	-1065	11	7	2	541	-489	10	1	3	876	813
15	1	2	1279	1386	-15	3	2	533	-420	6	4	2	703	688	1	8	2	424	447	11	1	3	454	408
16	1	2	703	665	-14	3	2	919	-903	7	4	2	889	860	2	8	2	680	665	12	1	3	508	-490
17	1	2	365	-313	-13	3	2	837	764	8	4	2	557	-529	5	8	2	405	-317	15	1	3	564	-593
-19	2	2	506	-322	-12	3	2	1212	1198	9	4	2	448	-455	6	8	2	632	-627	16	1	3	473	445
-18	2	2	640	454	-11	3	2	527	-450	10	4	2	664	633	8	8	2	1041	983	17	1	3	551	597
-15	2	2	797	790	-9	3	2	313	266	11	4	2	384	335	0	9	2	629	578	-18	2	3	402	-176
-14	2	2	825	-763	-8	3	2	1838	-1918	12	4	2	686	-627	1	9	2	281	261	-17	2	3	510	-338
-13	2	2	1663	-1647	-7	3	2	339	-366	0	5	2	1389	1288	-20	1	3	404	255	-16	2	3	711	720
-12	2	2	551	607	-6	3	2	1841	1906	2	5	2	486	-436	-18	1	3	379	238	-11	2	3	1696	-1779
-11	2	2	587	573	-5	3	2	250	74	4	5	2	690	-589	-16	1	3	1031	-986	-9	2	3	2767	2841
-10	2	2	741	768	-3	3	2	217	-153	6	5	2	1602	1542	-15	1	3	443	435	-8	2	3	474	475

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MOAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-7	2	3	2323	-2356	3	3	3	466	-446	15	4	3	407	-404	-1	6	3	1736	-1869	7	8	3	419	432
-4	2	3	678	-808	4	3	3	930	-990	-17	5	3	607	-546	0	6	3	416	-406	-4	9	3	565	513
-3	2	3	1732	1694	5	3	3	2283	-2260	-16	5	3	570	-504	3	6	3	848	861	-2	9	3	527	-447
-2	2	3	1665	1593	7	3	3	1991	1870	-14	5	3	790	748	4	6	3	1029	1039	-20	0	4	1165	1078
-1	2	3	1705	-1747	9	3	3	619	-599	-13	5	3	640	705	5	6	3	463	-455	-19	0	4	569	-493
0	2	3	1285	-1217	12	3	3	628	-576	-11	5	3	1071	-1119	6	6	3	545	-541	-18	0	4	514	-465
1	2	3	1642	-1627	13	3	3	554	464	-10	5	3	585	-580	7	6	3	567	-560	-16	0	4	958	-851
2	2	3	809	-852	14	3	3	806	792	-8	5	3	314	265	8	6	3	551	-513	-15	0	4	879	347
3	2	3	2980	3085	16	3	3	550	-582	-7	5	3	1139	1184	9	6	3	910	917	-14	0	4	1809	1911
4	2	3	1140	1146	-18	4	3	734	712	-6	5	3	777	822	10	6	3	941	917	-13	0	4	977	-1015
5	2	3	2831	-2777	-17	4	3	479	-501	-5	5	3	1161	-1161	11	6	3	949	-888	-10	0	4	4758	-5295
6	2	3	1082	-1033	-14	4	3	1231	-1319	-4	5	3	1471	-1478	12	6	3	531	-430	-9	0	4	352	-355
7	2	3	1005	997	-12	4	3	2043	2176	-3	5	3	315	-309	-12	7	3	592	423	-8	0	4	5726	6277
10	2	3	678	646	-10	4	3	771	-833	-2	5	3	1732	1766	-11	7	3	406	123	-7	0	4	783	742
15	2	3	627	776	-9	4	3	299	-135	-1	5	3	1822	1873	-10	7	3	484	237	-6	0	4	2999	-2816
16	2	3	578	565	-8	4	3	1837	-1938	0	5	3	914	-931	-8	7	3	401	-376	-5	0	4	256	204
-19	3	3	462	-590	-6	4	3	3249	3340	1	5	3	1520	-1599	-4	7	3	791	828	-4	0	4	2602	-2670
-18	3	3	682	604	-4	4	3	1928	-1997	2	5	3	1008	-1071	-3	7	3	282	220	-3	0	4	635	-625
-16	3	3	443	-338	-3	4	3	332	249	4	5	3	1112	1143	-2	7	3	1189	-1222	-2	0	4	2368	2326
-15	3	3	657	690	-2	4	3	1030	-1060	5	5	3	1211	1213	-1	7	3	443	-417	-1	0	4	681	629
-13	3	3	1721	-1808	-1	4	3	326	-287	7	5	3	429	-414	0	7	3	647	681	0	0	4	3037	-3097
-11	3	3	1380	1441	0	4	3	3220	3142	8	5	3	820	-772	1	7	3	378	464	1	0	4	1689	1642
-10	3	3	634	849	1	4	3	933	947	9	5	3	503	-474	2	7	3	299	271	2	0	4	566	-532
-9	3	3	1193	1259	2	4	3	1935	-2060	10	5	3	568	562	5	7	3	535	-483	3	0	4	1812	-1854
-8	3	3	1417	-1482	3	4	3	1000	-1035	11	5	3	802	739	6	7	3	352	-370	4	0	4	4393	4852
-7	3	3	2696	-2792	4	4	3	840	-872	13	5	3	619	-595	8	7	3	589	567	5	0	4	2308	2383
-6	3	3	1127	1166	6	4	3	2048	1981	-15	6	3	936	886	9	7	3	449	495	6	0	4	3265	-3400
-5	3	3	2237	2215	7	4	3	1270	1215	-14	6	3	409	-138	10	7	3	574	-524	7	0	4	1435	-1382
-3	3	3	498	-469	8	4	3	1213	-1185	-13	6	3	609	-558	-8	8	3	355	-310	8	0	4	586	615
-2	3	3	710	670	9	4	3	522	-476	-11	6	3	527	-490	-6	8	3	431	324	9	0	4	475	-490
-1	3	3	1651	-1801	11	4	3	326	-278	-9	6	3	1113	1182	-2	8	3	410	-312	10	0	4	848	791
0	3	3	1155	-1067	12	4	3	735	718	-7	6	3	393	-347	0	8	3	538	571	11	0	4	987	912
1	3	3	2669	2763	13	4	3	588	566	-5	6	3	1108	-1075	2	8	3	338	-380	12	0	4	560	-569
2	3	3	1957	2010	14	4	3	701	-703	-3	6	3	2220	2263	5	8	3	354	-322	13	0	4	719	-693

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MDAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC	H	K	L	10FO	10FC
16	0	4	466	453	-17	2	4	936	910	-16	3	4	628	-637	-8	4	4	282	-142	2	5	4	1082	-1054
-20	1	4	390	-196	-16	2	4	439	-411	-15	3	4	353	206	-7	4	4	551	-572	3	5	4	637	-651
-19	1	4	718	-613	-15	2	4	636	-594	-14	3	4	373	-304	-6	4	4	1244	1269	4	5	4	793	794
-18	1	4	792	777	-14	2	4	331	-163	-13	3	4	447	384	-5	4	4	840	895	8	5	4	677	-710
-15	1	4	1406	1028	-13	2	4	1436	-1479	-12	3	4	1599	1760	-4	4	4	1179	-1212	10	5	4	900	897
-14	1	4	482	-532	-12	2	4	414	334	-11	3	4	401	-405	-3	4	4	578	-543	11	5	4	336	-269
-13	1	4	1118	-1163	-11	2	4	2048	2990	-10	3	4	1732	-1789	-2	4	4	972	868	12	5	4	526	-473
-12	1	4	814	-813	-9	2	4	915	-924	-9	3	4	477	367	-1	4	4	395	385	13	5	4	569	581
-11	1	4	1037	-1100	-7	2	4	2327	-2399	-8	3	4	283	-386	0	4	4	1231	-1266	-16	6	4	500	-162
-10	1	4	1395	1402	-6	2	4	643	-582	-7	3	4	1700	-1669	1	4	4	341	-372	-15	6	4	449	24
-9	1	4	3622	3797	-5	2	4	2960	2881	-6	3	4	2294	2307	2	4	4	952	1024	-11	6	4	426	-499
-8	1	4	511	564	-4	2	4	847	315	-5	3	4	1762	1780	3	4	4	385	337	-10	6	4	344	-305
-7	1	4	2769	-2717	-3	2	4	2443	-2412	-4	3	4	2323	-2281	5	4	4	302	257	-9	6	4	462	545
-6	1	4	1375	-1314	-2	2	4	682	703	-3	3	4	319	296	8	4	4	571	-532	-5	6	4	1209	-1222
-5	1	4	360	-340	-1	2	4	207	-102	-2	3	4	1481	1440	9	4	4	525	-516	-3	6	4	934	944
-4	1	4	289	-256	0	2	4	1200	-1103	-1	3	4	468	-454	10	4	4	390	387	-2	6	4	393	357
-3	1	4	1556	1515	1	2	4	2827	2877	0	3	4	854	784	11	4	4	484	467	-1	6	4	585	549
-2	1	4	279	-217	2	2	4	1991	2001	1	3	4	418	405	13	4	4	335	-358	0	6	4	326	150
-1	1	4	1704	-1736	3	2	4	3146	-3291	2	3	4	2538	-2617	-17	5	4	641	-539	1	6	4	1126	-1155
0	1	4	534	-884	4	2	4	852	-879	4	3	4	1175	1198	-16	5	4	418	-200	2	6	4	696	-697
1	1	4	576	-524	5	2	4	478	496	5	3	4	650	-673	-15	5	4	732	661	3	6	4	505	449
2	1	4	1769	1804	6	2	4	973	-986	7	3	4	793	1036	-14	5	4	647	-688	4	6	4	539	550
3	1	4	2726	2796	7	2	4	1866	1875	8	3	4	548	-530	-13	5	4	659	-794	7	6	4	445	-449
4	1	4	414	415	8	2	4	1525	1544	9	3	4	1520	-1519	-12	5	4	891	900	9	6	4	589	561
5	1	4	3007	-3151	9	2	4	1269	-1311	10	3	4	596	543	-10	5	4	711	-724	-13	7	4	428	-373
6	1	4	1727	-1780	10	2	4	648	-676	13	3	4	431	417	-9	5	4	581	571	-11	7	4	412	-229
7	1	4	740	736	11	2	4	310	297	14	3	4	488	-475	-8	5	4	1300	1310	-10	7	4	335	139
8	1	4	1051	1016	12	2	4	407	-395	15	3	4	398	-399	-5	5	4	381	-379	-9	7	4	766	801
9	1	4	384	322	13	2	4	329	214	-18	4	4	440	443	-4	5	4	2059	-2131	-7	7	4	874	-920
11	1	4	429	-395	14	2	4	814	824	-17	4	4	404	27	-3	5	4	802	812	-6	7	4	337	-281
12	1	4	589	-584	15	2	4	362	-350	-16	4	4	611	-576	-2	5	4	850	836	-4	7	4	352	370
15	1	4	597	606	16	2	4	657	-638	-15	4	4	502	448	-1	5	4	1372	-1416	-3	7	4	1365	1476
-19	2	4	629	-552	-18	3	4	741	639	-14	4	4	1012	1060	0	5	4	1142	1179	-1	7	4	1440	-1472
-18	2	4	508	483	-17	3	4	452	-444	-12	4	4	878	-881	1	5	4	1695	1675	0	7	4	441	-398

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MOAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	7	4	424	370	-5	1	5	358	365	1	2	5	579	582	7	3	5	979	990	12	4	5	386	-308
3	7	4	404	425	-4	1	5	735	665	2	2	5	242	166	8	3	5	335	393	-17	5	5	558	-491
5	7	4	540	-510	-3	1	5	1358	-1305	3	2	5	1368	1355	9	3	5	1001	-1037	-16	5	5	532	-313
6	7	4	435	-478	-2	1	5	1476	1464	4	2	5	1049	1016	10	3	5	483	-481	-15	5	5	502	396
-11	8	4	444	288	-1	1	5	977	906	5	2	5	2742	-2881	11	3	5	476	498	-14	5	5	918	958
-8	8	4	550	-577	0	1	5	2973	-2879	6	2	5	1412	-1501	14	3	5	346	277	-12	5	5	1320	-1417
-6	8	4	450	239	1	1	5	415	-421	7	2	5	1917	2004	-18	4	5	963	959	-11	5	5	994	-981
-4	8	4	836	879	2	1	5	1438	1477	8	2	5	824	826	-17	4	5	449	-434	-10	5	5	452	407
-2	8	4	1421	-1466	3	1	5	2134	-2124	9	2	5	1003	-1042	-16	4	5	1170	-1256	-9	5	5	1125	1121
-1	8	4	481	-496	4	1	5	726	705	12	2	5	445	-407	-15	4	5	471	364	-8	5	5	1053	1065
0	8	4	847	831	5	1	5	2473	2559	13	2	5	308	200	-14	4	5	399	338	-6	5	5	847	-868
1	8	4	510	537	6	1	5	2137	-2184	-19	3	5	543	-544	-12	4	5	1228	1288	-5	5	5	977	-937
4	8	4	530	-540	7	1	5	952	-999	-18	3	5	801	823	-11	4	5	442	-437	-4	5	5	580	-568
6	8	4	321	228	8	1	5	1940	2042	-17	3	5	1053	1047	-10	4	5	2336	-2411	-3	5	5	832	778
7	8	4	361	227	9	1	5	413	-472	-16	3	5	611	-682	-9	4	5	589	-532	-2	5	5	1005	998
-6	9	4	476	450	10	1	5	581	-592	-15	3	5	780	-805	-8	4	5	984	981	-1	5	5	522	526
-4	9	4	550	-546	11	1	5	834	926	-14	3	5	380	402	-7	4	5	570	578	0	5	5	1352	-1318
-3	9	4	636	-679	12	1	5	320	-272	-11	3	5	2087	2119	-6	4	5	2003	1982	1	5	5	1680	-1661
-1	9	4	511	563	13	1	5	574	-556	-10	3	5	457	470	-5	4	5	230	149	2	5	5	452	493
0	9	4	611	602	14	1	5	376	454	-9	3	5	1937	-1983	-4	4	5	3242	-3275	3	5	5	1370	1304
2	9	4	471	-536	-19	2	5	858	-959	-8	3	5	990	-1005	-3	4	5	674	-684	4	5	5	656	652
-20	1	5	924	929	-17	2	5	618	567	-7	3	5	1358	-1344	-2	4	5	1797	1811	6	5	5	649	-680
-19	1	5	416	-404	-16	2	5	357	141	-6	3	5	1599	1536	0	4	5	802	793	7	5	5	940	-947
-18	1	5	692	-688	-13	2	5	1017	-1102	-5	3	5	3164	3201	1	4	5	534	539	9	5	5	464	490
-17	1	5	440	426	-10	2	5	330	243	-4	3	5	354	-319	2	4	5	2372	-2445	10	5	5	566	565
-14	1	5	963	999	-9	2	5	2139	2175	-3	3	5	2603	-2577	3	4	5	891	-848	-15	6	5	737	745
-13	1	5	431	-523	-7	2	5	3639	-3606	-2	3	5	365	-321	4	4	5	1751	1755	-14	6	5	435	-329
-12	1	5	1068	-1094	-6	2	5	637	-599	-1	3	5	731	713	5	4	5	976	984	-13	6	5	1293	-1353
-11	1	5	1267	1300	-5	2	5	3029	2931	1	3	5	1228	1215	6	4	5	334	330	-12	6	5	336	353
-10	1	5	1288	-1330	-4	2	5	203	46	2	3	5	1399	1454	7	4	5	430	414	-11	6	5	1023	1051
-9	1	5	1472	-1414	-3	2	5	376	-256	3	3	5	1795	-1759	8	4	5	1002	-1026	-9	6	5	328	302
-8	1	5	3487	3528	-2	2	5	561	542	4	3	5	1886	-1929	9	4	5	947	-958	-7	6	5	1236	-1246
-7	1	5	576	550	-1	2	5	1661	-1642	5	3	5	630	610	10	4	5	767	746	-6	6	5	727	-744
-6	1	5	4037	-3968	0	2	5	965	-932	6	3	5	769	787	11	4	5	514	552	-5	6	5	519	489

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MDAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC	H	K	L	10FO	10FC
-3	6	5	999	1034	3	8	5	303	-212	15	0	6	408	366	-9	2	6	3138	-3201	-1	3	6	910	856
-2	6	5	717	744	-5	9	5	334	-201	-19	1	6	1275	-1306	-8	2	6	337	-366	0	3	6	1079	-1029
-1	6	5	2125	-2164	0	9	5	390	410	-17	1	6	960	974	-7	2	6	2006	1973	1	3	6	384	-343
0	6	5	753	-742	-20	0	6	1738	1748	-16	1	6	691	738	-5	2	6	1238	1217	2	3	6	1429	-1494
1	6	5	1839	1886	-19	0	6	388	-375	-14	1	6	680	-595	-4	2	6	1069	1064	3	3	6	492	-456
2	6	5	533	558	-18	0	6	1932	-2064	-13	1	6	877	-852	-3	2	6	2805	-2787	4	3	6	2175	2155
3	6	5	395	-393	-16	0	6	854	922	-11	1	6	1090	1027	-2	2	6	704	-652	6	3	6	767	-729
4	6	5	343	337	-15	0	6	321	335	-10	1	6	1271	1259	-1	2	6	2401	2349	7	3	6	755	832
5	6	5	500	-484	-14	0	6	855	790	-9	1	6	1301	1334	0	2	6	501	480	9	3	6	1355	-1335
6	6	5	823	-810	-12	0	6	2037	-2128	-8	1	6	1583	-1528	1	2	6	476	-473	11	3	6	789	732
7	6	5	378	211	-11	0	6	582	641	-7	1	6	2808	-2765	2	2	6	538	546	-19	4	6	553	-409
8	6	5	320	324	-10	0	6	1000	957	-6	1	6	694	-630	3	2	6	2092	-2090	-17	4	6	405	238
10	6	5	477	497	-9	0	6	411	-396	-5	1	6	3119	2963	4	2	6	2188	-2159	-16	4	6	648	-652
11	6	5	416	-393	-8	0	6	3178	3128	-4	1	6	1637	1536	5	2	6	1845	1817	-14	4	6	949	979
-14	7	5	435	-579	-7	0	6	796	785	-3	1	6	1206	-1164	6	2	6	1213	1212	-13	4	6	452	328
-12	7	5	790	897	-6	0	6	5738	-5983	-2	1	6	1669	-1605	8	2	6	1003	1041	-12	4	6	1015	-1036
-10	7	5	523	-462	-5	0	6	1630	-1531	-1	1	6	1294	-1233	9	2	6	713	-724	-10	4	6	986	1028
-9	7	5	352	-425	-4	0	6	2676	2565	0	1	6	758	703	10	2	6	1146	-1192	-8	4	6	254	-234
-8	7	5	479	-406	-3	0	6	1947	1811	1	1	6	1378	1340	11	2	6	553	604	-5	4	6	755	726
-6	7	5	300	346	-2	0	6	637	623	2	1	6	1359	1330	12	2	6	359	345	-4	4	6	342	-348
-4	7	5	396	326	-1	0	6	792	-693	3	1	6	622	628	13	2	6	415	-405	-3	4	6	864	-874
-3	7	5	307	324	0	0	6	2579	-2554	4	1	6	1102	-1074	-20	3	6	679	680	-2	4	6	527	465
-2	7	5	581	-949	1	0	6	1557	-1559	5	1	6	2529	-2532	-19	3	6	468	-249	-1	4	6	696	707
-1	7	5	513	-524	2	0	6	2070	2053	6	1	6	842	-885	-17	3	6	559	509	0	4	6	676	-624
0	7	5	1252	1238	3	0	6	1407	1421	7	1	6	1706	1712	-16	3	6	1005	-1025	1	4	6	547	-532
1	7	5	440	385	4	0	6	494	491	8	1	6	1526	1534	-14	3	6	1148	1173	2	4	6	1052	1078
2	7	5	489	-496	5	0	6	914	855	10	1	6	549	-570	-10	3	6	1257	-1208	3	4	6	886	870
4	7	5	347	-334	6	0	6	2718	-2744	-20	2	6	392	44	-8	3	6	1683	1632	4	4	6	644	-642
5	7	5	311	-390	7	0	6	2515	-2592	-17	2	6	1041	1081	-7	3	6	425	-382	7	4	6	766	-720
7	7	5	334	256	8	0	6	1514	1528	-15	2	6	1505	-1667	-6	3	6	519	-465	8	4	6	331	-360
-11	8	5	519	-386	9	0	6	1023	1062	-13	2	6	1048	1016	-5	3	6	1750	1667	11	4	6	461	439
-10	8	5	438	-458	10	0	6	585	-659	-12	2	6	337	-335	-4	3	6	1172	-1093	-18	5	6	405	252
-5	8	5	355	328	11	0	6	549	546	-11	2	6	1219	1220	-3	3	6	1761	-1719	-16	5	6	869	-826
2	8	5	328	-334	13	0	6	556	-580	-10	2	6	948	976	-2	3	6	2032	1999	-15	5	6	468	479

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR NDAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-14	5	6	741	781	0	6	6	236	-175	-4	9	6	455	-468	-13	2	7	296	2	-7	3	7	2174	2194
-13	5	6	682	-675	1	6	6	327	-338	-3	9	6	535	-504	-12	2	7	408	-422	-6	3	7	1738	1705
-11	5	6	825	851	3	6	6	543	497	-2	9	6	514	445	-11	2	7	1501	1496	-5	3	7	487	449
-10	5	6	925	-895	5	6	6	370	-369	-1	9	6	568	652	-10	2	7	806	792	-4	3	7	1028	-997
-9	5	6	790	-825	9	6	6	514	515	-20	1	7	855	924	-9	2	7	460	-380	-3	3	7	2118	-2106
-8	5	6	572	941	-14	7	6	524	-293	-18	1	7	1550	-1650	-8	2	7	336	-340	-1	3	7	2062	2044
-7	5	6	295	297	-13	7	6	810	-939	-16	1	7	1479	1505	-7	2	7	1734	-1734	0	3	7	1156	1108
-6	5	6	365	-336	-11	7	6	898	839	-15	1	7	479	-537	-6	2	7	969	-933	1	3	7	706	-724
-4	5	6	1014	-1006	-7	7	6	737	-735	-14	1	7	674	-659	-5	2	7	3450	3418	2	3	7	399	-389
-3	5	6	292	-251	-6	7	6	373	-317	-13	1	7	818	826	-4	2	7	2057	2001	4	3	7	866	-843
-2	5	6	1510	1503	-5	7	6	718	769	-12	1	7	1287	-1266	-3	2	7	2931	-2928	5	3	7	850	826
-1	5	6	524	-508	-4	7	6	645	638	-11	1	7	603	-556	-2	2	7	984	-963	6	3	7	1910	1852
0	5	6	1000	-953	-1	7	6	1080	-1097	-10	1	7	2409	2411	-1	2	7	1497	1471	7	3	7	434	-376
1	5	6	1323	1316	0	7	6	537	-556	-9	1	7	961	-960	0	2	7	196	68	8	3	7	912	-931
3	5	6	1192	-1189	1	7	6	1042	1087	-8	1	7	883	-856	1	2	7	495	431	9	3	7	436	-401
4	5	6	659	626	2	7	6	546	531	-7	1	7	2501	2439	2	2	7	662	646	11	3	7	404	475
5	5	6	500	447	3	7	6	611	-579	-6	1	7	2124	-2060	3	2	7	778	-765	12	3	7	427	429
6	5	6	646	-689	4	7	6	311	-282	-5	1	7	2182	-2158	4	2	7	283	-226	-19	4	7	426	12
10	5	6	719	742	7	7	6	310	326	-4	1	7	3613	3512	5	2	7	673	-621	-16	4	7	1167	-1210
12	5	6	651	-668	8	7	6	354	309	-3	1	7	633	604	6	2	7	813	-809	-14	4	7	1860	1929
-15	6	6	693	634	-11	8	6	398	344	-2	1	7	1962	-1898	7	2	7	1440	1423	-12	4	7	1046	-1022
-13	6	6	412	-302	-10	8	6	662	-590	-1	1	7	364	422	8	2	7	1162	1187	-11	4	7	470	-420
-12	6	6	341	-376	-9	8	6	673	-640	1	1	7	1061	-1017	9	2	7	1065	-1107	-10	4	7	389	-850
-11	6	6	473	-416	-7	8	6	357	257	2	1	7	1019	1039	10	2	7	852	-865	-8	4	7	2457	2457
-10	6	6	302	-118	-6	8	6	654	604	3	1	7	1180	1152	11	2	7	433	436	-7	4	7	1375	1390
-9	6	6	646	647	-5	8	6	353	313	4	1	7	1054	-992	-18	3	7	527	577	-6	4	7	1515	-1445
-8	6	6	741	710	-4	8	6	340	-200	7	1	7	1322	-1324	-17	3	7	711	726	-5	4	7	1229	-1246
-7	6	6	360	-376	-2	8	6	648	-661	8	1	7	842	831	-16	3	7	836	-872	-4	4	7	852	-850
-6	6	6	829	-805	-1	8	6	361	-171	9	1	7	1008	985	-15	3	7	1501	-1590	-3	4	7	644	-680
-5	6	6	323	-274	0	8	6	1038	991	10	1	7	1048	-1073	-14	3	7	351	385	-2	4	7	1959	1886
-4	6	6	271	238	1	8	6	443	494	12	1	7	493	464	-13	3	7	1502	1529	-1	4	7	1564	1573
-3	6	6	775	808	2	8	6	557	-546	-19	2	7	1035	-1069	-11	3	7	474	-431	0	4	7	1532	-1487
-2	6	6	385	238	3	8	6	440	-410	-17	2	7	1656	1765	-9	3	7	1874	-1813	1	4	7	972	-945
-1	6	6	555	-594	-5	9	6	385	-72	-15	2	7	1155	-1155	-8	3	7	1266	-1195	4	4	7	1230	1201

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MOAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
5	4	7	1140	1100	-6	6	7	453	-418	-14	0	8	1325	-1349	-11	1	8	936	901	-5	2	8	2705	-2672
6	4	7	1075	-1025	-5	6	7	1039	1010	-13	0	8	316	-225	-9	1	8	1376	-1350	-4	2	8	693	-671
7	4	7	1080	-1041	-4	6	7	917	942	-12	0	8	280	-105	-8	1	8	1403	-1406	-3	2	8	961	-915
10	4	7	411	449	-3	6	7	388	-335	-11	0	8	317	-323	-7	1	8	574	-582	-2	2	8	1726	-1691
11	4	7	651	616	-1	6	7	640	-625	-10	0	8	2638	2813	-6	1	8	1703	1598	-1	2	8	2190	2181
-15	5	7	573	531	0	6	7	963	-956	-9	0	8	470	435	-5	1	8	3318	3279	0	2	8	1835	1818
-14	5	7	627	470	1	6	7	1533	1512	-8	0	8	3337	-3280	-4	1	8	1731	1679	1	2	8	1561	-1558
-13	5	7	530	-560	2	6	7	1037	1006	-7	0	8	659	-586	-3	1	8	2896	-2828	2	2	8	713	-700
-12	5	7	1223	-1281	3	6	7	1416	-1477	-6	0	8	1320	-1254	-2	1	8	3071	-3043	3	2	8	367	393
-11	5	7	317	-309	4	6	7	824	-812	-5	0	8	2265	-2142	-1	1	8	873	842	4	2	8	436	-500
-10	5	7	1403	1444	5	6	7	424	346	-4	0	8	4561	4685	0	1	8	1403	1318	5	2	8	448	434
-9	5	7	1284	1309	8	6	7	512	468	-3	0	8	3290	3309	1	1	8	598	543	6	2	8	1670	1613
-8	5	7	306	37	-13	7	7	340	-190	-2	0	8	3253	-3313	2	1	8	301	309	7	2	8	603	-593
-7	5	7	927	-923	-12	7	7	739	755	-1	0	8	1885	-1859	3	1	8	290	-208	8	2	8	845	-799
-6	5	7	1663	-1660	-10	7	7	1138	-1148	0	0	8	588	568	4	1	8	1036	-994	9	2	8	426	365
-5	5	7	285	-273	-9	7	7	547	-592	1	0	8	504	-478	6	1	8	471	486	12	2	8	564	540
-4	5	7	1022	1055	-8	7	7	443	490	2	0	8	605	530	7	1	8	925	829	13	2	8	350	-247
-3	5	7	842	813	-7	7	7	326	249	3	0	8	2198	2182	8	1	8	649	670	-20	3	8	867	937
-2	5	7	537	515	-5	7	7	417	425	4	0	8	1636	-1668	9	1	8	687	-652	-18	3	8	769	-743
-1	5	7	524	-486	-3	7	7	354	-365	5	0	8	1565	-1525	10	1	8	1089	-1033	-17	3	8	773	776
0	5	7	1103	-1080	-1	7	7	417	-332	7	0	8	336	-320	12	1	8	602	577	-15	3	8	1188	-1270
1	5	7	335	-374	0	7	7	563	552	8	0	8	610	612	-19	2	8	1339	1327	-14	3	8	1171	1242
2	5	7	938	957	1	7	7	511	490	9	0	8	1710	1622	-18	2	8	339	-93	-13	3	8	526	463
3	5	7	967	966	2	7	7	805	-800	10	0	8	961	-915	-17	2	8	335	-154	-12	3	8	1808	-1834
4	5	7	518	-463	3	7	7	517	-471	11	0	8	1247	-1241	-16	2	8	310	334	-10	3	8	821	789
5	5	7	739	-704	-10	8	7	370	-273	12	0	8	566	620	-15	2	8	1064	-1096	-8	3	8	1784	1793
6	5	7	383	-360	-8	8	7	575	578	-20	1	8	559	-586	-14	2	8	440	-419	-7	3	8	330	300
8	5	7	341	401	-3	8	7	413	-422	-19	1	8	768	-744	-13	2	8	1939	1900	-6	3	8	2857	-2844
9	5	7	676	679	-1	8	7	341	264	-17	1	8	1572	1620	-11	2	8	2227	-2232	-5	3	8	293	-272
-13	6	7	1075	-1055	-20	0	8	639	507	-16	1	8	363	313	-10	2	8	276	165	-4	3	8	1445	1399
-11	6	7	1912	1960	-18	0	8	2062	-2144	-15	1	8	1182	-1215	-9	2	8	493	-447	-3	3	8	1424	-1433
-10	6	7	342	270	-17	0	8	368	-363	-14	1	8	692	-691	-8	2	8	1202	-1209	-2	3	8	710	661
-9	6	7	1433	-1406	-16	0	8	2520	2543	-13	1	8	556	576	-7	2	8	3747	3817	-1	3	8	2617	2627
-8	6	7	385	-424	-15	0	6	618	677	-12	1	8	817	821	-6	2	8	1905	1839	0	3	8	918	-857

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MOAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	3	8	1718	-1725	-13	5	8	420	-248	6	6	8	337	-253	-15	1	9	418	421	-7	2	9	650	632
2	3	8	663	693	-12	5	8	1345	-1354	7	6	8	346	293	-14	1	9	1553	-1501	-6	2	9	308	-288
4	3	8	379	342	-11	5	8	633	704	8	6	8	358	250	-12	1	9	758	687	-5	2	9	1929	1861
5	3	8	818	837	-9	5	8	1526	-1518	-13	7	8	453	-456	-11	1	9	759	-722	-4	2	9	1761	1704
6	3	8	686	-869	-8	5	8	670	634	-11	7	8	1168	1217	-10	1	9	1233	1233	-3	2	9	2868	-2844
7	3	8	737	-695	-7	5	8	1331	1349	-9	7	8	993	-1011	-9	1	9	999	971	-2	2	9	2175	-2147
8	3	8	639	619	-6	5	8	973	-969	-8	7	8	462	-442	-8	1	9	2227	-2172	-1	2	9	2492	2466
10	3	8	383	-417	-5	5	8	421	-423	-5	7	8	344	341	-7	1	9	226	161	0	2	9	1255	1222
11	3	8	510	552	-4	5	8	736	759	-4	7	8	394	274	-6	1	9	1231	1168	1	2	9	881	-906
-15	4	8	393	-220	-1	5	8	352	337	-3	7	8	605	-542	-5	1	9	2127	-2081	3	2	9	290	-229
-17	4	8	400	370	0	5	8	1472	-1475	-2	7	8	449	-482	-4	1	9	1386	1353	4	2	9	571	-495
-15	4	8	535	-592	1	5	8	252	-167	0	7	8	279	234	-3	1	9	2315	2263	5	2	9	828	775
-14	4	8	518	528	2	5	8	1472	1506	1	7	8	502	552	-2	1	9	2467	-2954	8	2	9	590	558
-13	4	8	614	623	3	5	8	461	-443	2	7	6	512	538	-1	1	9	693	-663	9	2	9	406	-497
-12	4	8	395	-379	4	5	8	550	-548	3	7	8	817	-807	0	1	9	2103	2113	10	2	9	918	-892
-10	4	8	845	843	5	5	8	971	952	4	7	8	839	-786	1	1	9	668	-687	11	2	9	551	487
-9	4	8	348	241	7	5	8	854	-821	5	7	8	475	453	2	1	9	679	-735	12	2	9	729	683
-8	4	8	1328	-1340	8	5	8	400	362	6	7	8	474	463	3	1	9	1111	1081	-19	3	9	987	968
-7	4	8	598	-612	9	5	8	375	371	-10	8	8	557	-569	4	1	9	576	-572	-17	3	9	520	-562
-4	4	8	460	439	-15	6	8	433	334	-9	8	8	695	-605	5	1	9	1160	-1106	-16	3	9	854	-885
-3	4	8	257	258	-13	6	8	924	-963	-8	8	8	555	538	6	1	9	791	778	-15	3	9	765	-773
-2	4	8	450	485	-12	6	8	435	-366	-7	8	8	577	575	8	1	9	346	-324	-14	3	9	755	709
-1	4	8	378	325	-11	6	8	458	404	-4	8	8	513	-457	9	1	9	957	924	-13	3	9	1746	1731
0	4	8	666	-645	-9	6	8	529	489	-3	8	8	450	-400	11	1	9	1038	-1006	-11	3	9	1721	-1674
1	4	8	594	-594	-8	6	8	548	478	-2	8	8	371	337	12	1	9	637	603	-10	3	9	801	-759
2	4	8	324	324	-7	6	8	591	-574	-1	8	8	411	442	-20	2	9	403	-114	-9	3	9	840	802
3	4	8	679	707	-6	6	8	698	-686	0	8	8	254	273	-17	2	9	1427	1502	-7	3	9	1327	1246
5	4	8	474	-450	-5	6	8	438	387	2	8	8	657	-691	-16	2	9	450	430	-6	3	9	1456	1453
6	4	8	560	563	-4	6	8	647	577	3	8	8	467	-509	-15	2	9	2256	-2312	-5	3	9	1985	-1981
8	4	8	589	-583	-1	6	8	799	-794	-20	1	9	505	-462	-14	2	9	427	-427	-4	3	9	2047	-2034
10	4	8	390	389	0	6	8	366	-331	-19	1	9	757	752	-13	2	9	1718	1739	-3	3	9	248	176
-18	5	8	547	-459	1	6	8	816	845	-18	1	9	749	-744	-10	2	9	359	312	-2	3	9	820	800
-16	5	8	381	-249	2	6	8	323	272	-17	1	9	773	-718	-9	2	9	1628	-1550	-1	3	9	1284	1261
-14	5	8	1268	1286	5	6	8	433	-441	-16	1	9	1826	1888	-8	2	9	293	-243	0	3	9	514	460

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MDAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	3	9	1448	-1443	-16	5	9	754	843	2	6	9	706	729	10	0	10	583	513	-4	1	10	711	-665
2	3	9	964	-956	-15	5	9	333	-84	3	6	9	795	-785	-9	0	10	550	526	-3	1	10	2267	-2168
3	3	9	746	765	-14	5	9	393	-443	4	6	9	1210	-1156	-8	0	10	2358	-2273	-2	1	10	2184	-2116
4	3	9	576	534	-13	5	9	500	-526	5	6	9	648	655	-7	0	10	2280	-2226	-1	1	10	1550	1523
6	3	9	734	712	-12	5	9	357	-333	6	6	9	961	898	-6	0	10	2079	1978	0	1	10	2012	2024
8	3	9	1440	-1382	-11	5	9	542	531	-12	7	9	358	163	-5	0	10	1188	1150	1	1	10	354	-352
10	3	9	881	867	-10	5	9	1239	1220	-11	7	9	303	267	-4	0	10	1483	1342	2	1	10	867	-853
-18	4	9	1195	-1212	-9	5	9	338	307	-10	7	9	742	-777	-3	0	10	2184	2134	5	1	10	614	584
-17	4	9	377	-442	-8	5	9	1299	-1305	-8	7	9	1041	1002	-2	0	10	2759	-2721	6	1	10	633	572
-16	4	9	820	511	-7	5	9	1266	-1277	-7	7	9	516	519	-1	0	10	2759	-2803	8	1	10	343	-359
-14	4	9	1091	1137	-5	5	9	868	866	-5	7	9	306	-209	0	0	10	2358	2332	9	1	10	369	-431
-13	4	9	411	431	-4	5	9	1355	1378	-4	7	9	414	-412	1	0	10	1246	1206	10	1	10	418	-441
-12	4	9	2130	-2144	-3	5	9	349	310	-3	7	9	355	-343	2	0	10	1144	-1239	11	1	10	500	425
-11	4	9	657	-593	-2	5	9	765	-771	1	7	9	378	395	5	0	10	1093	-1047	-20	2	10	374	-299
-10	4	9	1371	1369	-1	5	9	995	-945	2	7	9	351	-364	6	0	10	621	592	-19	2	10	888	907
-9	4	9	356	409	0	5	9	693	-663	3	7	9	585	-541	7	0	10	997	948	-17	2	10	1814	-1847
-8	4	9	626	570	1	5	9	612	602	4	7	9	697	668	8	0	10	399	-319	-15	2	10	719	686
-7	4	9	481	425	2	5	9	1018	1030	5	7	9	486	466	10	0	10	492	-403	-13	2	10	1052	1041
-6	4	9	2423	-2384	4	5	9	688	-694	-10	8	9	326	162	11	0	10	1357	-1290	-12	2	10	282	182
-5	4	9	1650	-1669	5	5	9	688	-685	-8	8	9	331	121	-19	1	10	483	461	-11	2	10	2250	-2213
-4	4	9	1685	1677	7	5	9	749	691	-6	8	9	446	-330	-18	1	10	834	768	-10	2	10	781	-783
-3	4	9	1073	1083	8	5	9	359	334	-5	8	9	375	-328	-17	1	10	408	355	-9	2	10	2080	2056
-1	4	9	538	557	-16	6	9	348	222	-3	8	9	360	106	-16	1	10	473	-487	-8	2	10	692	652
0	4	9	1309	-1274	-15	6	9	763	-800	-1	8	9	401	379	-15	1	10	1859	-1867	-7	2	10	734	682
1	4	9	1729	-1738	-11	6	9	973	1003	1	8	9	399	-418	-14	1	10	784	-765	-6	2	10	1212	1198
2	4	9	1316	1375	-10	6	9	470	411	-20	0	10	1197	-1232	-13	1	10	1941	1899	-5	2	10	2438	-2416
3	4	9	1020	1003	-9	6	9	2023	-2099	-18	0	10	634	557	-12	1	10	2069	2029	-4	2	10	2108	-2161
4	4	9	496	-499	-8	6	9	722	-745	-17	0	10	356	-106	-11	1	10	882	-897	-3	2	10	1606	1537
5	4	9	304	269	-7	6	9	1518	1545	-16	0	10	1767	1764	-10	1	10	1379	-1381	-2	2	10	425	363
6	4	9	399	-401	-6	6	9	592	626	-15	0	10	563	616	-9	1	10	1015	-985	-1	2	10	421	377
7	4	9	1260	-1207	-4	6	9	236	215	-14	0	10	2838	-2771	-8	1	10	208	-183	0	2	10	1264	1251
8	4	9	653	560	-3	6	9	665	-676	-13	0	10	1545	-1513	-7	1	10	1372	1269	1	2	10	1063	-1073
9	4	9	1029	1011	-2	6	9	859	-805	-12	0	10	2118	2046	-6	1	10	1916	1899	2	2	10	1110	-1102
-17	5	9	653	634	-1	6	9	309	378	-11	0	10	1531	1563	-5	1	10	965	971	3	2	10	970	979

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MLAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	2	10	617	548	-13	4	10	428	450	1	5	10	341	-331	-7	8	10	561	528	-12	2	11	1562	1520
5	2	10	536	-539	-11	4	10	393	-379	2	5	10	970	1014	-6	8	10	638	-595	-11	2	11	2058	-2014
8	2	10	957	-878	-10	4	10	346	261	3	5	10	488	487	-5	8	10	615	613	-10	2	11	1239	-1212
9	2	10	452	395	-9	4	10	323	351	4	5	10	997	-963	-2	8	10	468	423	-9	2	11	661	649
10	2	10	514	858	-8	4	10	838	-859	6	5	10	545	491	-1	8	10	418	404	-8	2	11	456	395
-18	3	10	870	-857	-7	4	10	966	-998	7	5	10	729	-710	0	8	10	345	-272	-7	2	11	946	941
-16	3	10	1320	1329	-6	4	10	1045	1065	-16	6	10	629	589	-20	1	11	907	-915	-6	2	11	446	416
-15	3	10	430	-357	-5	4	10	436	403	-11	6	10	651	716	-18	1	11	1296	1312	-5	2	11	506	-506
-14	3	10	523	-489	-4	4	10	434	-417	-9	6	10	318	-368	-17	1	11	708	-722	-4	2	11	448	-456
-13	3	10	1184	1177	-3	4	10	626	631	-5	6	10	590	550	-15	1	11	1272	1265	-3	2	11	858	-868
-12	3	10	830	-809	-2	4	10	295	309	-3	6	10	464	-345	-14	1	11	1626	1629	-2	2	11	894	-857
-11	3	10	1180	-1181	-1	4	10	548	-467	-2	6	10	595	-586	-13	1	11	1118	-1113	-1	2	11	1485	1520
-10	3	10	1469	1424	0	4	10	492	-482	0	6	10	272	295	-12	1	11	2079	2034	0	2	11	1804	1826
-9	3	10	955	951	1	4	10	337	-378	1	6	10	859	863	-11	1	11	296	414	1	2	11	877	-902
-8	3	10	334	-300	3	4	10	463	437	2	6	10	384	363	-10	1	11	1515	-1493	2	2	11	1247	-1292
-7	3	10	345	-317	5	4	10	407	-435	3	6	10	602	-618	-9	1	11	560	540	3	2	11	272	325
-6	3	10	1688	-1683	6	4	10	318	346	4	6	10	325	-303	-7	1	11	1626	-1572	4	2	11	404	313
-5	3	10	830	-771	7	4	10	634	625	-14	7	10	377	-291	-6	1	11	1454	1393	-18	3	11	337	197
-4	3	10	1971	1947	8	4	10	460	-411	-13	7	10	630	533	-5	1	11	1336	1305	-17	3	11	1249	-1239
-3	3	10	586	538	-18	5	10	611	-482	-12	7	10	398	308	-4	1	11	1094	-1056	-16	3	11	729	-736
-2	3	10	755	-684	-17	5	10	506	-626	-11	7	10	419	181	-3	1	11	824	747	-15	3	11	1083	1068
-1	3	10	1075	1058	-16	5	10	532	562	-9	7	10	903	-895	-2	1	11	410	-325	-14	3	11	943	961
0	3	10	315	271	-12	5	10	779	-788	-8	7	10	545	-513	-1	1	11	1832	-1850	-13	3	11	378	282
1	3	10	1371	-1402	-11	5	10	279	-138	-7	7	10	984	1003	0	1	11	1476	1483	-12	3	11	651	-594
2	3	10	279	215	-10	5	10	1272	1318	-6	7	10	702	696	1	1	11	1232	1268	-11	3	11	1582	1577
3	3	10	675	632	-9	5	10	525	-459	-5	7	10	556	-533	2	1	11	1142	-1193	-10	3	11	646	-624
4	3	10	653	-613	-8	5	10	878	-808	-4	7	10	576	-537	4	1	11	757	727	-9	3	11	1797	1742
7	3	10	455	-428	-7	5	10	1039	1085	-1	7	10	550	562	6	1	11	332	-141	-8	3	11	1190	1149
8	3	10	756	723	-5	5	10	949	-959	0	7	10	678	670	7	1	11	510	452	-7	3	11	1208	-1201
9	3	10	686	633	-4	5	10	614	573	1	7	10	276	-268	-19	2	11	581	545	-6	3	11	295	-296
10	3	10	455	-388	-3	5	10	557	596	2	7	10	320	-367	-17	2	11	352	-229	-5	3	11	638	-570
-18	4	10	378	335	-2	5	10	794	-766	4	7	10	477	-481	-15	2	11	1364	-1356	-4	3	11	1849	-1813
-15	4	10	323	-341	-1	5	10	577	-502	-10	8	10	547	-468	-14	2	11	960	-980	-3	3	11	1310	1281
-14	4	10	314	-129	0	5	10	280	-291	-8	8	10	769	775	-13	2	11	2077	2094	-2	3	11	2201	2230

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR ROAD

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-1	3	11	395	-314	-16	5	11	575	533	-7	7	11	486	403	5	0	12	551	544	-13	2	12	915	-900
0	3	11	939	-937	-15	5	11	617	-576	-6	7	11	793	-822	6	0	12	379	-228	-11	2	12	881	-876
1	3	11	644	-700	-14	5	11	1185	-1171	-5	7	11	684	-712	7	0	12	643	576	-10	2	12	1247	-1195
2	3	11	437	-434	-12	5	11	468	487	-4	7	11	328	123	9	0	12	635	-625	-9	2	12	1445	1396
3	3	11	868	889	-11	5	11	463	453	-3	7	11	498	427	-20	1	12	646	643	-8	2	12	1492	1439
4	3	11	757	723	-10	5	11	521	557	-2	7	11	343	338	-19	1	12	595	535	-7	2	12	1601	-1561
5	3	11	504	-506	-9	5	11	723	-690	-1	7	11	385	292	-18	1	12	381	278	-6	2	12	977	-974
6	3	11	426	-365	-8	5	11	1271	-1255	-4	8	11	459	327	-17	1	12	1134	-1081	-5	2	12	688	670
7	3	11	336	228	-6	5	11	1311	1364	-3	8	11	426	347	-16	1	12	1167	-1189	-4	2	12	722	-688
8	3	11	393	-399	-5	5	11	1066	1073	-20	0	12	475	-431	-15	1	12	697	-684	-3	2	12	751	739
-18	4	11	610	-522	-3	5	11	756	-710	-19	0	12	347	-247	-13	1	12	1989	1973	-2	2	12	1855	1857
-17	4	11	498	-487	-2	5	11	900	-900	-18	0	12	2177	2197	-12	1	12	1903	1872	-1	2	12	1054	-1036
-16	4	11	1418	1384	0	5	11	515	506	-17	0	12	651	796	-11	1	12	1785	-1723	0	2	12	1248	-1277
-15	4	11	1168	1215	1	5	11	635	639	-16	0	12	1500	-1526	-10	1	12	2258	-2226	1	2	12	281	272
-14	4	11	1164	-1128	2	5	11	454	488	-15	0	12	290	118	-9	1	12	808	792	2	2	12	290	-311
-13	4	11	507	-491	3	5	11	359	-311	-14	0	12	1439	-1423	-8	1	12	1062	1012	4	2	12	786	784
-12	4	11	400	-331	4	5	11	781	-761	-13	0	12	1782	-1734	-7	1	12	511	457	5	2	12	295	-278
-11	4	11	737	-726	6	5	11	336	458	-12	0	12	3152	3141	-6	1	12	401	266	6	2	12	893	-851
-10	4	11	1657	1604	7	5	11	536	558	-11	0	12	2832	2788	-5	1	12	1138	-1126	8	2	12	376	307
-9	4	11	1876	1546	-14	6	11	356	-291	-10	0	12	2656	-2567	-4	1	12	1414	-1344	-20	3	12	924	-926
-8	4	11	1454	-1461	-13	6	11	735	772	-9	0	12	1451	-1404	-2	1	12	547	564	-19	3	12	358	-270
-7	4	11	1092	-1069	-11	6	11	392	-347	-8	0	12	1282	1259	-1	1	12	948	933	-16	3	12	1474	1431
-5	4	11	330	-383	-10	6	11	347	271	-7	0	12	1286	-1207	0	1	12	1214	1244	-15	3	12	597	598
-4	4	11	1351	1330	-9	6	11	694	-624	-6	0	12	699	621	1	1	12	974	-1028	-14	3	12	1772	-1740
-3	4	11	1841	1823	-8	6	11	624	-626	-5	0	12	2798	2776	2	1	12	1623	-1694	-12	3	12	642	622
-2	4	11	593	-569	-7	6	11	1554	1593	-4	0	12	1719	-1604	3	1	12	359	383	-11	3	12	1422	-1435
-1	4	11	1503	-1556	-6	6	11	1140	1162	-3	0	12	1909	-1855	4	1	12	1109	1071	-9	3	12	1716	1677
2	4	11	556	529	-5	6	11	1088	-1132	-2	0	12	507	457	8	1	12	552	-545	-8	3	12	760	-734
3	4	11	1118	1108	-4	6	11	1044	-1043	-1	0	12	1220	-1216	-20	2	12	337	-34	-7	3	12	973	-974
4	4	11	657	-637	-1	6	11	456	416	0	0	12	1357	1348	-19	2	12	1034	-1028	-6	3	12	540	509
5	4	11	891	-846	0	6	11	625	616	1	0	12	2701	2846	-17	2	12	1014	-1031	-4	3	12	545	534
6	4	11	372	295	4	6	11	345	-410	2	0	12	1321	-1386	-16	2	12	674	-676	-3	3	12	1006	981
-18	5	11	638	616	-12	7	11	357	-205	3	0	12	1989	-2029	-15	2	12	2376	2353	-2	3	12	982	-990
-17	5	11	608	501	-8	7	11	532	557	4	0	12	950	976	-14	2	12	1073	1044	-1	3	12	1032	-981

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MOAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	3	12	736	724	0	5	12	239	323	-6	1	13	721	-703	6	2	13	479	-426	-5	4	13	1003	982
2	3	12	478	-523	1	5	12	474	460	-5	1	13	1301	1252	-14	3	13	1262	-1281	-4	4	13	367	-388
3	3	12	624	636	5	5	12	423	-347	-4	1	13	553	-480	-18	3	13	342	-262	-3	4	13	756	747
5	3	12	500	-837	6	5	12	527	536	-3	1	13	1468	-1433	-17	3	13	395	381	-2	4	13	826	-812
7	3	12	517	504	-14	6	12	611	-538	-2	1	13	1005	995	-15	3	13	1173	1174	-1	4	13	1738	-1733
-18	4	12	843	861	-13	6	12	323	361	0	1	13	368	-349	-14	3	13	1230	1247	0	4	13	616	618
-16	4	12	445	-488	-12	6	12	464	501	1	1	13	1519	1611	-13	3	13	1383	-1353	1	4	13	1189	1243
-13	4	12	380	-244	-9	6	12	775	-803	2	1	13	512	-495	-12	3	13	1305	-1310	5	4	13	828	-824
-10	4	12	608	495	-8	6	12	518	-493	3	1	13	1554	-1547	-10	3	13	603	591	-18	5	13	916	915
-9	4	12	462	467	-7	6	12	658	684	4	1	13	815	805	-9	3	13	1324	1305	-16	5	13	839	-857
-8	4	12	387	-354	-6	6	12	572	541	5	1	13	695	696	-8	3	13	675	630	-15	5	13	789	-746
-7	4	12	821	-841	-3	6	12	450	-346	6	1	13	716	-674	-7	3	13	1515	-1574	-14	5	13	347	-403
-6	4	12	314	257	0	6	12	464	487	-20	2	13	442	346	-6	3	13	1280	-1314	-13	5	13	609	570
-5	4	12	682	646	-13	7	12	407	334	-17	2	13	770	-729	-5	3	13	1012	1021	-12	5	13	1046	1054
-4	4	12	507	-936	-11	7	12	780	-773	-16	2	13	358	-197	-2	3	13	1329	1373	-10	5	13	326	-306
-3	4	12	558	-551	-10	7	12	483	-475	-15	2	13	318	266	-1	3	13	506	-486	-9	5	13	678	-686
-2	4	12	688	759	-7	7	12	618	666	-14	2	13	351	-302	0	3	13	1868	-1969	-8	5	13	896	-795
0	4	12	449	-453	-6	7	12	701	691	-13	2	13	1080	1113	2	3	13	748	762	-7	5	13	777	770
2	4	12	292	305	-5	7	12	843	-798	-12	2	13	1261	1230	3	3	13	364	326	-6	5	13	1262	1234
5	4	12	347	-345	-4	7	12	938	-863	-11	2	13	1892	-1880	5	3	13	554	-479	-4	5	13	848	-804
7	4	12	477	441	-2	7	12	459	482	-10	2	13	1900	-1879	6	3	13	595	-526	-3	5	13	1125	-1093
-17	5	12	899	-878	0	7	12	259	192	-9	2	13	1659	1630	-19	4	13	452	-440	-1	5	13	783	777
-15	5	12	784	722	-19	1	13	678	-610	-8	2	13	1110	1138	-18	4	13	881	980	0	5	13	554	553
-14	5	12	716	-635	-18	1	13	746	705	-7	2	13	585	-561	-17	4	13	481	367	4	5	13	403	-423
-13	5	12	319	-278	-17	1	13	616	588	-5	2	13	1008	-961	-16	4	13	508	538	-16	6	13	523	-584
-12	5	12	613	601	-16	1	13	1481	-1466	-4	2	13	631	-592	-15	4	13	640	643	-15	6	13	823	829
-10	5	12	681	646	-15	1	13	351	299	-3	2	13	810	801	-14	4	13	1581	-1597	-14	6	13	668	646
-9	5	12	529	478	-14	1	13	445	420	-2	2	13	481	456	-13	4	13	1437	-1431	-13	6	13	374	220
-8	5	12	1492	-1523	-13	1	13	1192	-1227	0	2	13	653	652	-12	4	13	1418	1404	-11	6	13	566	-643
-6	5	12	1214	1245	-12	1	13	1345	1336	1	2	13	595	-629	-11	4	13	774	731	-10	6	13	483	-457
-5	5	12	544	-566	-11	1	13	1489	1433	2	2	13	1333	-1362	-9	4	13	978	950	-9	6	13	494	503
-4	5	12	401	-432	-10	1	13	1988	-1941	3	2	13	450	486	-8	4	13	1558	-1554	-6	6	13	663	693
-3	5	12	1005	995	-9	1	13	776	-750	4	2	13	1101	1102	-7	4	13	1667	-1683	-5	6	13	951	-1005
-1	5	12	684	-883	-8	1	13	1872	1940	5	2	13	430	-386	-6	4	13	1612	1618	-4	6	13	1279	-1262

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MOAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-3	6	13	667	685	-18	1	14	755	-770	-8	2	14	570	520	6	3	14	441	-411	1	5	14	570	526
-2	6	13	1023	1072	-17	1	14	810	-785	-7	2	14	880	-862	-19	4	14	366	-243	2	5	14	283	-341
0	6	13	329	-22	-16	1	14	607	-615	-6	2	14	974	-954	-17	4	14	501	466	3	5	14	460	-431
2	6	13	475	-521	-15	1	14	935	862	-5	2	14	1186	1144	-16	4	14	761	-782	-15	6	14	379	-85
-8	7	13	322	44	-14	1	14	970	938	-4	2	14	1476	1459	-15	4	14	426	-464	-13	6	14	328	269
-6	7	13	461	-462	-13	1	14	430	464	-3	2	14	366	-194	-14	4	14	692	698	-12	6	14	419	428
-5	7	13	494	-509	-11	1	14	1364	-1301	-1	2	14	519	-544	-12	4	14	410	-328	-10	6	14	639	-650
-4	7	13	717	636	-10	1	14	1624	-1579	0	2	14	1603	-1667	-11	4	14	422	405	-9	6	14	427	-447
-3	7	13	763	727	-9	1	14	1388	1315	1	2	14	250	280	-10	4	14	326	289	-7	6	14	634	700
-20	0	14	1180	1160	-8	1	14	1804	1784	2	2	14	1043	1082	-8	4	14	285	-325	-6	6	14	524	471
-19	0	14	397	426	-7	1	14	878	-883	6	2	14	670	-663	-7	4	14	311	-345	-5	6	14	407	-296
-17	0	14	785	852	-6	1	14	1087	-1086	-20	3	14	491	-354	-5	4	14	401	363	1	6	14	311	-232
-16	0	14	1775	-1684	-5	1	14	721	-738	-19	3	14	543	-563	-4	4	14	435	-379	-11	7	14	471	-403
-15	0	14	1562	-1585	-4	1	14	277	-253	-18	3	14	659	584	-3	4	14	943	-944	-10	7	14	465	-378
-14	0	14	1141	1100	-3	1	14	1164	1160	-17	3	14	591	591	-2	4	14	501	548	-9	7	14	668	695
-12	0	14	1002	964	-2	1	14	1240	1207	-14	3	14	1004	-969	-1	4	14	923	987	-8	7	14	662	694
-11	0	14	1741	1704	-1	1	14	287	-151	-13	3	14	825	-830	0	4	14	297	-280	-5	7	14	369	-293
-10	0	14	2440	-2372	0	1	14	396	-387	-12	3	14	1197	1157	2	4	14	359	387	-4	7	14	598	-608
-9	0	14	2199	-2147	1	1	14	910	-953	-11	3	14	573	479	4	4	14	462	-464	-3	7	14	559	394
-8	0	14	2318	2347	2	1	14	1046	-1094	-10	3	14	927	-859	-17	5	14	409	-269	-20	1	15	1119	1077
-7	0	14	1631	1626	3	1	14	529	558	-9	3	14	562	536	-16	5	14	589	-637	-19	1	15	410	366
-6	0	14	1183	-1182	4	1	14	1129	1097	-8	3	14	428	352	-15	5	14	582	553	-18	1	15	996	-936
-5	0	14	274	-188	6	1	14	347	-405	-7	3	14	539	-928	-13	5	14	769	-727	-17	1	15	453	414
-4	0	14	605	-594	-19	2	14	1243	-1222	-6	3	14	623	622	-12	5	14	584	580	-15	1	15	1212	-1200
-3	0	14	1614	-1575	-18	2	14	623	-665	-5	3	14	1039	1036	-11	5	14	781	826	-14	1	15	994	956
-2	0	14	1046	1072	-17	2	14	1024	1004	-4	3	14	590	-606	-10	5	14	370	-347	-13	1	15	571	573
-1	0	14	1438	1451	-16	2	14	428	266	-3	3	14	449	-371	-8	5	14	324	-340	-12	1	15	700	-841
1	0	14	596	1052	-15	2	14	442	515	-2	3	14	529	-516	-7	5	14	642	-659	-11	1	15	844	785
2	0	14	649	-639	-14	2	14	843	849	-1	3	14	827	-857	-6	5	14	932	982	-10	1	15	632	-601
3	0	14	2210	-2219	-13	2	14	1609	-1568	0	3	14	648	666	-5	5	14	458	486	-9	1	15	1673	-1642
4	0	14	649	657	-12	2	14	1240	-1220	1	3	14	1003	1067	-4	5	14	995	-974	-8	1	15	1066	1027
5	0	14	1439	1453	-11	2	14	708	660	2	3	14	297	-225	-3	5	14	425	372	-7	1	15	1366	1374
6	0	14	480	-484	-10	2	14	350	294	4	3	14	320	280	-2	5	14	726	708	-6	1	15	1208	1167
-20	1	14	1029	1011	-9	2	14	331	274	5	3	14	702	-693	-1	5	14	471	-597	-5	1	15	695	-689

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MCAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FC	10FC	H	K	L	10FO	10FC
-4	1	15	802	778	-11	3	15	1092	1071	-11	5	15	596	-543	-6	0	16	1533	-1488	-15	2	16	1373	-1357
-3	1	15	316	-202	-10	3	15	1646	1584	-10	5	15	863	-863	-5	0	16	2387	-2378	-14	2	16	814	-792
-1	1	15	756	767	-8	3	15	499	-561	-8	5	15	354	205	-4	0	16	611	564	-12	2	16	617	-657
0	1	15	545	-313	-7	3	15	1016	-1030	-7	5	15	550	527	-3	0	16	433	517	-11	2	16	1093	1062
2	1	15	352	317	-6	3	15	523	-445	-6	5	15	624	590	-1	0	16	949	968	-10	2	16	1912	1880
3	1	15	759	-727	-5	3	15	1146	1124	-5	5	15	467	-407	1	0	16	908	-869	-9	2	16	842	-791
5	1	15	571	925	-4	3	15	1007	1001	-4	5	15	859	-838	3	0	16	360	-309	-8	2	16	1076	-1076
-20	2	15	624	901	-3	3	15	728	-739	-3	5	15	418	-320	5	0	16	946	917	-6	2	16	325	-234
-19	2	15	830	-865	-2	3	15	500	-472	-2	5	15	567	484	-20	1	16	876	940	-4	2	16	1290	1258
-18	2	15	917	-917	0	3	15	653	-693	-1	5	15	981	1020	-19	1	16	974	-977	-2	2	16	1328	-1379
-15	2	15	698	654	2	3	15	1188	1176	1	5	15	694	-716	-18	1	16	1082	-1062	2	2	16	871	882
-14	2	15	676	618	4	3	15	553	-556	-14	6	15	1078	1067	-17	1	16	519	478	3	2	16	334	274
-13	2	15	390	-390	-18	4	15	1236	1243	-13	6	15	647	-606	-16	1	16	736	783	4	2	16	678	-692
-12	2	15	625	-487	-17	4	15	937	934	-12	6	15	585	-973	-15	1	16	822	790	-19	3	16	659	-655
-11	2	15	522	-573	-16	4	15	1024	-1087	-8	6	15	644	674	-14	1	16	365	399	-17	3	16	736	753
-10	2	15	875	-869	-15	4	15	738	-735	-6	6	15	392	-295	-13	1	16	1282	-1261	-16	3	16	609	-525
-9	2	15	720	641	-13	4	15	770	-716	-4	6	15	362	-290	-12	1	16	1230	-1208	-15	3	16	748	-755
-8	2	15	1798	1818	-12	4	15	959	993	-3	6	15	440	401	-10	1	16	389	328	-14	3	16	517	504
-7	2	15	702	-676	-11	4	15	1784	1826	-2	6	15	950	960	-9	1	16	1205	1188	-13	3	16	324	213
-6	2	15	1495	-1466	-10	4	15	893	-823	-1	6	15	363	-278	-8	1	16	1369	1392	-12	3	16	447	346
-5	2	15	292	252	-9	4	15	1175	-1178	-20	0	16	1607	1650	-7	1	16	959	-980	-11	3	16	967	963
-4	2	15	485	464	-7	4	15	314	-290	-19	0	16	1344	1233	-6	1	16	1863	-1846	-10	3	16	760	-744
-2	2	15	403	364	-6	4	15	754	763	-18	0	16	1814	-1797	-4	1	16	901	892	-9	3	16	1310	-1335
0	2	15	476	-476	-5	4	15	1414	1404	-17	0	16	774	-740	-3	1	16	465	494	-8	3	16	593	572
3	2	15	315	316	-4	4	15	877	-872	-16	0	16	734	706	-2	1	16	364	228	-6	3	16	560	-579
4	2	15	666	660	-3	4	15	1032	-1039	-15	0	16	807	-787	-1	1	16	332	-374	-5	3	16	813	827
-19	3	15	973	-954	0	4	15	379	375	-14	0	16	1253	1250	0	1	16	791	-793	-3	3	16	1200	-1214
-18	3	15	534	-472	1	4	15	929	980	-13	0	16	2073	2057	1	1	16	377	-332	-2	3	16	326	39
-17	3	15	1441	1515	3	4	15	711	-718	-12	0	16	1504	-1491	3	1	16	394	418	-1	3	16	576	670
-16	3	15	756	701	-17	5	15	517	-508	-11	0	16	742	-706	4	1	16	496	500	1	3	16	340	304
-15	3	15	614	-584	-16	5	15	1124	-1141	-10	0	16	278	-320	-19	2	16	344	-148	3	3	16	490	-491
-14	3	15	377	329	-14	5	15	950	954	-9	0	16	1725	-1743	-18	2	16	572	-516	-18	4	16	325	58
-13	3	15	892	-798	-13	5	15	958	918	-8	0	16	1424	1443	-17	2	16	966	937	-17	4	16	512	447
-12	3	15	1505	-1518	-12	5	15	328	171	-7	0	16	2925	2942	-16	2	16	1078	1050	-15	4	16	423	-408

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-14	4	16	777	753	-13	1	17	1188	1171	-16	3	17	631	616	-9	5	17	645	630	-9	1	18	833	587
-13	4	16	741	675	-12	1	17	792	-751	-15	3	17	1387	-1408	-8	5	17	668	591	-7	1	18	845	-854
-12	4	16	841	-852	-11	1	17	951	-980	-14	3	17	891	-877	-6	5	17	359	-173	-6	1	18	1106	-1103
-11	4	16	410	-341	-10	1	17	937	945	-13	3	17	804	786	-4	5	17	475	-358	-5	1	18	546	524
-10	4	16	461	395	-9	1	17	532	-529	-11	3	17	342	327	-2	5	17	601	574	-4	1	18	1033	1012
-5	4	16	352	339	-8	1	17	337	-233	-10	3	17	1435	1470	-10	6	17	879	925	-2	1	18	644	-621
-4	4	16	458	-410	-7	1	17	1770	1771	-9	3	17	521	-537	-7	6	17	321	55	-19	2	18	554	516
-3	4	16	604	-537	-6	1	17	575	-402	-8	3	17	1410	-1405	-20	0	18	583	584	-18	2	18	532	477
-1	4	16	826	805	-5	1	17	1744	-1711	-6	3	17	663	607	-19	0	18	1005	1003	-16	2	18	384	265
1	4	16	607	-600	-4	1	17	758	776	-5	3	17	747	731	-18	0	18	1596	-1581	-15	2	18	781	-727
-16	5	16	949	-906	-3	1	17	1006	959	-4	3	17	360	330	-17	0	18	1639	-1635	-14	2	18	1463	-1466
-14	5	16	792	760	-2	1	17	813	-830	-3	3	17	771	-716	-16	0	18	1648	1596	-13	2	18	344	339
-13	5	16	427	-411	0	1	17	444	467	-2	3	17	761	-782	-15	0	18	1695	1678	-12	2	18	1307	1301
-12	5	16	597	-543	1	1	17	705	-707	0	3	17	621	625	-12	0	18	1033	-986	-10	2	18	597	609
-11	5	16	837	807	3	1	17	472	441	-17	4	17	604	582	-11	0	18	1813	-1815	-9	2	18	432	-394
-9	5	16	834	-832	-20	2	17	801	838	-16	4	17	1256	-1252	-10	0	18	750	669	-8	2	18	1778	-1788
-8	5	16	406	400	-19	2	17	852	-853	-15	4	17	1145	-1113	-9	0	18	755	675	-7	2	18	846	167
-7	5	16	423	359	-18	2	17	1131	-1111	-14	4	17	1064	1026	-8	0	18	302	236	-6	2	18	964	868
-5	5	16	321	290	-17	2	17	894	815	-13	4	17	773	722	-7	0	18	1436	1424	-5	2	18	413	-149
-4	5	16	377	-358	-16	2	17	1018	1023	-12	4	17	303	58	-6	0	18	605	-598	-2	2	18	576	-595
-3	5	16	571	-525	-14	2	17	722	-656	-11	4	17	762	676	-5	0	18	2177	-2159	0	2	18	838	875
-2	5	16	633	726	-12	2	17	774	-772	-10	4	17	648	-644	-4	0	18	499	497	-18	3	18	654	-636
0	5	16	503	-538	-11	2	17	672	583	-9	4	17	1541	-1595	-3	0	18	1567	1532	-15	3	18	725	-699
-10	6	16	464	-450	-10	2	17	710	661	-8	4	17	536	555	-2	0	18	326	-382	-14	3	18	594	537
-9	6	16	350	141	-8	2	17	624	604	-7	4	17	1036	1023	-1	0	18	641	-605	-13	3	18	1079	1086
-8	6	16	578	582	-7	2	17	356	-325	-6	4	17	340	-140	-19	1	18	860	-853	-11	3	18	344	-263
-5	6	16	375	-255	-6	2	17	1632	-1613	-3	4	17	1170	-1120	-16	1	18	682	-719	-10	3	18	418	-407
-4	6	16	390	-305	-5	2	17	575	562	-2	4	17	385	486	-17	1	18	1029	1009	-9	3	18	1094	-1055
-20	1	17	637	670	-4	2	17	1345	1342	-1	4	17	951	1024	-16	1	18	1201	1239	-8	3	18	947	907
-19	1	17	917	909	-3	2	17	439	-463	-15	5	17	418	432	-14	1	18	992	-940	-6	3	18	335	-229
-18	1	17	1190	-1197	-2	2	17	427	-485	-14	5	17	954	931	-13	1	18	1084	-1091	-4	3	18	731	722
-17	1	17	606	-805	0	2	17	389	-415	-13	5	17	439	474	-12	1	18	473	-478	-3	3	18	720	-683
-16	1	17	1246	1206	2	2	17	448	463	-12	5	17	725	-667	-11	1	18	1052	1023	-2	3	18	341	-345
-14	1	17	530	-517	-17	3	17	1023	1010	-11	5	17	1082	-1099	-10	1	18	1209	1163	-1	3	18	738	786

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MDAU

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-14	4	18	314	155	-2	1	19	349	-208	-14	4	19	824	819	-11	1	20	646	606	-13	1	21	1423	-1433
-13	4	18	600	601	-1	1	19	638	-698	-13	4	19	1120	1095	-9	1	20	436	-208	-12	1	21	553	584
-12	4	18	421	-390	-18	2	19	641	-676	-12	4	19	653	-662	-8	1	20	671	-638	-11	1	21	875	844
-11	4	18	1123	-1123	-17	2	19	533	484	-11	4	19	1140	-1084	-7	1	20	529	-427	-10	1	21	734	-712
-9	4	18	643	632	-16	2	19	1241	1264	-9	4	19	316	-149	-5	1	20	574	494	-8	1	21	459	323
-6	4	18	420	358	-15	2	19	526	-504	-8	4	19	373	358	-4	1	20	482	415	-7	1	21	934	-862
-4	4	18	400	-321	-14	2	19	1148	-1134	-7	4	19	924	972	-3	1	20	360	-263	-5	1	21	525	464
-3	4	18	335	-372	-12	2	19	333	296	-5	4	19	852	-826	-2	1	20	618	-678	-15	2	21	330	-334
-12	5	18	640	-827	-10	2	19	506	576	-18	0	20	448	-404	-17	2	20	407	-362	-14	2	21	1376	-1356
-10	5	18	590	598	-8	2	19	619	-635	-17	0	20	1199	-1196	-16	2	20	820	-764	-13	2	21	540	495
-9	5	18	391	-339	-4	2	19	1052	1009	-16	0	20	877	859	-14	2	20	489	-320	-12	2	21	1268	1251
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-5	5	18	508	-517	-16	3	19	501	-427	-14	0	20	435	-535	-10	2	20	1304	-1279	-8	2	21	561	-470
-19	1	19	565	576	-15	3	19	836	-838	-13	0	20	2014	-1962	-9	2	20	369	250	-6	2	21	480	506
-17	1	19	1172	-1130	-14	3	19	337	-459	-12	0	20	411	360	-8	2	20	1102	1098	-12	3	21	509	362
-16	1	19	632	565	-13	3	19	1022	1053	-11	0	20	522	488	-4	2	20	840	-789	-11	3	21	633	-672
-15	1	19	1397	1359	-12	3	19	1087	1074	-9	0	20	1136	1122	-14	3	20	437	-430	-10	3	21	1115	-1069
-14	1	19	872	-877	-11	3	19	546	-550	-7	0	20	963	-886	-13	3	20	907	889	-13	0	22	1522	-1480
-13	1	19	708	-737	-10	3	19	544	-456	-5	0	20	413	-301	-11	3	20	1230	-1209	-12	0	22	337	338
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-9	1	19	846	847	-5	3	19	421	290	-16	1	20	957	921	-11	4	20	560	-557	-13	1	22	485	410
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-5	1	19	893	-925	-15	4	19	441	-338	-12	1	20	800	799	-14	1	21	379	-214	-9	1	22	494	-475
-3	1	19	983	1014																				

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Publications:

1) Deesterification of Pectins and Methods of Analysis. Technical Report at H.J. Heinz. E.M. Kinsch, 1981

2) A ^{31}P Nuclear Magnetic Resonance and Fluorescence Study of the Interaction of an Anti-Arthritic Gold Phosphine Drug with Albumin. A Bioinorganic Approach. E. M. Kinsch and D.W. Stephan. Inorganica Chimica Acta, 91 263-267, 1984.

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by E. M. Kinsch and D.W. Stephan.
67th Annual CIC Conference, Montreal 1984.